REDACTED VERSION

TARGET SHEET

SITE NAME: CEDAR CHEMICAL

CERCLIS I.D.: ARD990660649

TITLE OF DOC.: PROCESS DESCRIPTIONS FOR THE PRODUCTION OF NITROPARAFFIN DERIVATIVES

DATE OF DOC.: 09/21/1987

NO. OF PGS. THIS TARGET SHEET REPLACES: 81

SDMS #: 9547856 **RELATED #:** 9351895

SENSITIVE ?	X	**MISSING PAGES ?**	
ALTERN. MEDIA ?		**CROSS REFERENCE ?**	

LAB DOCUMENT ?		**LAB NAME:**	
ASC./BOX #:			
CASE #:		**SDG #:**	

COMMENTS : PAGES 1-81 WERE REDACTED FROM THIS DOCUMENT DUE TO FOIA EXEMPTION B(4) - CONFIDENTIAL BUSINESS INFORMATION.

9547856

CEDAR CHEMICAL CORPORATION

24th Floor • 5100 Poplar Avenue • Memphis, TN 38137 • 901-685-5348

February 11, 1992

Mr. Edward G. Najjar, President
Organic Chemicals Division
W. R. Grace & Company
55 Hayden Avenue
Lexington, MA 02173

Dear Mr. Najjar:

Under paragraph 9 (d) of our Nitroparaffin Derivatives Contract, the base fees are escalated at the beginning of each contract year according to a formula. The calculations and support documents are attached for each of the three types of production months for the next contract year. The new fees will commence on January 26, 1992, the first day of the third contract year.

We notice that on page 28 of the Contract, notices are to be directed to Mr. William J. Eissler at Cedar. We propose to change this to Geoffrey L. Pratt, Director of Custom Manufacturing, Cedar Chemical Corporation, 24th Floor, Clark Tower, 5100 Poplar Ave., Memphis, TN 38137.

If the above changes meet with your approval, would you please initial below and return a copy of this letter to my attention.

We feel that our two companies have made significant improvements in the derivatives operation during 1991 and look forward to increased productivity and reduced costs in our third contract year. We continue to appreciate your business.

Sincerely,

Geoffrey L. Pratt
Director of Custom Manufacturing

mc
Enclosure

cc: Richard Zagraniczny

bcc: R. Tomblin
N. Robbins
D. Hoppel
Official File

APPROVED: _____
W. R. Grace & Company

CEDAR - WEST HELENA
BASE LEVEL ADJUSTMENT (GRACE)
FOR 3ND YEAR. 1992

COST	COST INDICATORS		Year 1991 Base Year RATIO OF INDICATORS	% TO TOTAL	1ST YEAR 1990	% TO TOTAL	3rd YEAR 1992
HOURLY WAGE	$ HR. BASE	Year 1991	10.93	53.0%	74.200	54.3%	81.920
		Base Year	9.90				
ELECTRICITY	COST PER KWH	Year 1991	0.057049	6.0%	8.400	5.2%	7.818
		Base Year	0.061295				
GAS	COST PER MCF	Year 1991	2.677018	3.0%	4.200	2.4%	3.626
		Base Year	3.101011				
PLANT COST	U.S. CONSUMER PRICE INDEX		137.8	38.0%	53.200	38.1%	57.543
			127.4				
TOTAL				100.0%	140.000	100.0%	150.905

MONTHLY PRODUCTION FEE		140.000
NEW YEAR CALCULATION	150.905	
LESS BASE LEVEL PER CONTRACT SCHEDULE	-140.000	
1992 MONTHLY PRODUCTION FEE		150.905

CEDAR - WEST HELENA
BASE LEVEL ADJUSTMENT (GRACE) START-UP
FOR 3ND YEAR. 1992

COST	COST INDICATORS		Year 1991 Base Year RATIO OF INDICATORS	% TO TOTAL	1ST YEAR 1990	% TO TOTAL	3rd YEAR 1992
HOURLY WAGE	$ HR. BASE	Year 1991	10.93	53.0%	92.750	54.3%	102.400
		Base Year	9.90				
ELECTRICITY	COST PER KWH	Year 1991	0.057049	6.0%	10.500	5.2%	9.773
		Base Year	0.061295				
GAS	COST PER MCF	Year 1991	2.677018	3.0%	5.250	2.4%	4.532
		Base Year	3.101011				
PLANT COST	U.S. CONSUMER PRICE INDEX		137.8 / 127.4	38.0%	66.500	38.1%	71.929
TOTAL				100.0%	175.000	100.0%	188.633

MONTHLY START-UP FEE		175.000
NEW YEAR CALCULATION	188.633	
LESS BASE LEVEL PER CONTRACT SCHEDULE	-175.000	
1992 MONTHLY START-UP FEE		188.633

CEDAR - WEST HELENA
BASE LEVEL ADJUSTMENT (GRACE) IDLE PLANT
FOR 3ND YEAR. 1992

COST	COST INDICATORS	Year 1991 Base Year RATIO OF INDICATORS		% TO TOTAL	1ST YEAR 1990	% TO TOTAL	3rd YEAR 1992
HOURLY WAGE	$ HR. BASE	Year 1991	10.93	53.0%	53.000	54.3%	58.514
		Base Year	9.90				
ELECTRICITY	COST PER KWH	Year 1991	0.057049	6.0%	6.000	5.2%	5.584
		Base Year	0.061295				
GAS	COST PER MCF	Year 1991	2.677018	3.0%	3.000	2.4%	2.590
		Base Year	3.101011				
PLANT COST	U.S. CONSUMER PRICE INDEX		137.8	38.0%	38.000	38.1%	41.102
			127.4				
TOTAL				100.0%	100.000	100.0%	107.790
MONTHLY IDLE PLANT FEE					100.000		
NEW YEAR CALCULATION					107.790		
LESS BASE LEVEL PER CONTRACT SCHEDULE					-100.000		
1992 MONTHLY IDLE PLANT FEE					107.790		

CALCULATIONS

MONTH	GAS MCF USED	TOTAL COST	COST MCF
BASE YEAR 1989			
Jan-89	6.144	19.463.52	3.167891
Feb-89	7.682	23.911.11	3.112615
Mar-89	6.612	20.600.37	3.115603
Apr-89	5.034	15.684.22	3.115658
May-89	3.687	11.422.64	3.098085
Jun-89	3.271	10.133.56	3.098001
Jul-89	2.016	6.323.17	3.136493
Aug-89	2.361	7.388.83	3.129534
Sep-89	2.255	7.060.45	3.13102
Oct-89	2.138	6.354.36	2.972105
Nov-89	2.693	8.067.95	2.995897
Dec-89	2.830	8.478.34	2.99588
TOTAL	46.723	144.888.52	3.10101
2nd YEAR 1991			
Jan-91	6.930	19.378.13	2.796267
Feb-91	8.760	23.560.67	2.689574
Mar-91	8.032	22.097.45	2.751177
Apr-91	7.377	20.547.36	2.785327
May-91	6.974	19.127.84	2.742736
Jun-91	7.388	20.113.22	2.722417
Jul-91	6.668	18.454.68	2.767648
Aug-91	8.759	23.248.43	2.654233
Sep-91	7.863	21.186.76	2.694488
Oct-91	9.201	24.247.71	2.635334
Nov-91	10.986	28.088.01	2.556709
Dec-91	13.510	34.204.93	2.531823
TOTAL	102.448	274.255.19	2.67702

METER NO	MONTH	ELECTRICITY KWH USED	TOTAL COST	COST KWH
BASE YEAR 1989				
39-131-284	Dec-88	277.200	15.995.10	0.057702
	Jan-89	253.400	15.166.82	0.059853
	Feb-89	284.200	16.712.57	0.058806
	Mar-89	282.800	15.476.47	0.054726
	Apr-89	250.600	14.764.02	0.058915
	May-89	267.400	17.546.39	0.065619
	Jun-89	417.200	23.224.53	0.055668
	Jul-89	102.200	11.125.79	0.108863
	Aug-89	323.400	19.995.19	0.061828
	Sep-89	443.800	26.852.93	0.060507
	Oct-89	305.200	16.410.66	0.053770
	Nov-89	224.000	12.612.88	0.056308
70-758-960/	Dec-88	6.785	362.21	0.053384
42-707-716	Jan-89	6.300	305.88	0.048552
	Feb-89	6.772	345.10	0.050960
	Mar-89	6.640	319.43	0.048107
	Apr-89	6.773	345.14	0.050958
	May-89	8.557	814.75	0.095214
	Jun-89	10.492	986.12	0.093988
	Jul-89	9.528	892.03	0.093622
	Aug-89	12.328	1.140.68	0.092528
	Sep-89	8.261	768.76	0.093059
	Oct-89	8.126	353.44	0.043495
	Nov-89	8.355	427.32	0.051145
42-795-109/	Nov-88	2.928	176.16	0.060164
81-895-094	Dec-88	2.557	144.89	0.056664
	Jan-89	2.970	170.94	0.057556
	Feb-89	2.581	145.66	0.056435
	Mar-89	1.955	124.46	0.063662
	Apr-89	2.597	253.48	0.097605
	May-89	3.696	353.16	0.095552
	Jun-89	3.938	373.91	0.094949
	Jul-89	5.009	468.78	0.093588
	Sep-89	2.940	279.35	0.095017
	Oct-89	2.994	152.28	0.050862
	Nov-89	4.003	222.91	0.055686
41-978-537/	Dec-88	119.760	7.015.28	0.058578
41-978-541/	Jan-89	78.760	4.629.63	0.058781
49-353-409	Feb-89	106.800	6.180.90	0.057874
	Mar-89	113.000	6.021.68	0.053289
	Apr-89	91.760	5.352.14	0.058328
	May-89	94.080	5.782.44	0.061463
	Jun-89	73.680	4.761.28	0.064621

METER NO	MONTH	ELECTRICITY KWH USED	TOTAL COST	COST KWH
2nd Year 1991				
2-700-416	Dec-90	625.800	32.989.91	0.052716
	Jan-91	544.600	30.170.42	0.055399
	Feb-91	526.400	28.682.98	0.054489
	Mar-91	620.200	33.495.67	0.054008
	Apr-91	777.000	41.151.18	0.052962
	May-91	805.000	43.898.23	0.054532
	Jun-91	824.600	51.093.30	0.061961
	Jul-91	862.400	52.043.71	0.060348
	Aug-91	849.800	54.937.92	0.064648
	Sep-91	866.600	52.884.11	0.061025
	Oct-91	953.400	48.877.61	0.051267
	Nov-91	919.800	47.593.98	0.051744
	Dec-91	837.200	44.070.74	0.052641
70-758-960	Dec-90	6.685	363.09	0.054314
	Jan-91	7.357	400.33	0.054415
	Feb-91	6.074	326.34	0.053727
	Mar-91	6.422	348.99	0.054343
	Apr-91	6.794	369.23	0.054346
	May-91	1.577	158.22	0.100330
81-895-094	Dec-90	4.761	268.74	0.056446
	Jan-91	7.912	427.87	0.054079
	Feb-91	5.373	292.76	0.054487
	Mar-91	5.213	285.08	0.054686
49-353-409	Dec-90	76.200	5.022.83	0.065916
	Jan-91	123.360	7.356.44	0.059634
	Feb-91	155.640	8.219.54	0.052811
	Mar-91	165.960	8.881.57	0.053516
	Apr-91	155.160	8.478.07	0.054641
	May-91	117.840	7.155.72	0.060724
	Jun-91	125.280	7.820.91	0.062427
	Jul-91	131.160	8.326.07	0.063480
	Aug-91	137.520	8.680.16	0.063119
	Sep-91	146.040	8.703.80	0.059599
	Oct-91	124.800	7.272.89	0.058276
	Nov-91	145.680	7.720.23	0.052994
	Dec-91	173.400	9.734.59	0.056140
39-153-684	Dec-90	5.920	517.66	0.087443
	Jan-91	5.560	446.41	0.080290
	Feb-91	6.200	506.09	0.081627
	Mar-91	5.600	487.08	0.086979
	Apr-91	7.400	610.26	0.082468
	May-91	8.200	757.10	0.092329
	Jun-91	10.040	890.38	0.088683
	Jul-91	12.960	1.072.30	0.082739
	Aug-91	12.360	1.050.81	0.085017
	Sep-91	11.280	907.52	0.080454

ELECTRICITY

METER NO	MONTH	KWH USED	TOTAL COST	COST KWH
BASE YEAR 1991				
	Jul-89	61.240	3.997.56	0.065277
	Aug-89	50.280	3.171.04	0.063068
	Sep-89	68.680	4.607.07	0.067080
	Oct-89	67.080	3.839.95	0.057244
	Nov-89	41.720	2.585.46	0.061972
55-210-742	Dec-88	8.773	462.31	0.052697
	Jan-89	7.065	342.99	0.048548
	Feb-89	7.704	391.98	0.050880
	Mar-89	6.891	334.79	0.048584
	Apr-89	7.571	385.88	0.050968
	May-89	10.059	960.39	0.095476
	Jun-89	4.157	400.30	0.096295
	Jul-89	22.681	2.115.33	0.093264
	Aug-89	14.558	1.349.61	0.092706
	Sep-89	13.357	1.241.70	0.092962
	Oct-89	5.480	253.92	0.046336
	Nov-89	6.633	350.64	0.052863
55-053-007/	Dec-88	18.680	1.404.66	0.075196
42-657-049	Jan-89	19.160	1.362.43	0.071108
	Feb-89	17.480	1.306.11	0.074720
	Mar-89	19.640	1.344.77	0.068471
	Apr-89	21.600	1.488.27	0.068901
	May-89	25.440	1.908.99	0.075039
	Jun-89	24.400	1.792.20	0.073451
	Jul-89	23.680	1.705.17	0.072009
	Aug-89	24.960	1.739.31	0.069684
	Sep-89	12.600	1.254.73	0.099582
	Oct-89	16.320	1.004.86	0.061572
	Nov-89	17.680	1.193.37	0.067498
		4.891.894	299.849.33	0.061295

ELECTRICITY Cont'd

METER NO	MONTH	KWH USED	TOTAL COST	COST KWH
2nd Year 1991				
	Oct-91	10.080	785.21	0.077898
	Nov-91	9.640	765.51	0.079410
	Dec-91	7.040	568.56	0.080761
61-349-019	Jan-91	12.200	828.41	0.067902
	Feb-91	11.080	760.57	0.068644
	Mar-91	5.480	501.23	0.091465
	Apr-91	3.120	445.66	0.142840
	May-91	800	560.50	0.700625
	Jun-91	680	562.81	0.827662
	Jul-91	680	562.19	0.826750
	Aug-91	960	563.28	0.586750
	Sep-91	1.560	522.54	0.334962
	Oct-91	1.560	419.75	0.269071
	Nov-91	1.840	420.65	0.228614
	Dec-91	9.160	478.17	0.052202
75-767-973	Jan-91	5.920	648.96	0.109622
	Feb-91	6.880	675.07	0.098121
	Mar-91	160	98.24	0.614000
		12.023.368	685.916.15	0.057049

CHARLES METCALF CRUMP
JOHN B. MAXWELL, JR.
ALLEN T. MALONE
PHILIP G. KAMINSKY
ROBERT L. DINKELSPIEL
HENRY L. KLEIN
ROBERT J. PINSTEIN
JOHN L. RYDER
THOMAS R. BUCKNER
BRUCE M. SMITH
*TONI CAMPBELL PARKER
STEVEN N. DOUGLASS
G. COBLE CAPERTON
RANDY S. GARDNER
LINDA D. SCHOLL
JANE P. LONG
**DAVID F. FREUDIGER
DAVID W. HAWKINS
RICHARD J. MYERS
ANN M. TUCKER

*ALSO ADMITTED IN MISSISSIPPI
**ALSO ADMITTED IN DISTRICT OF COLUMBIA

LAW OFFICES
APPERSON, CRUMP & MAXWELL, *PLC*
SUITE 2110
ONE COMMERCE SQUARE
MEMPHIS, TENNESSEE 38103-2519
901 / 525-1711

FACSIMILE 901 / 521-0789

EAST OFFICE:
SUITE 100
1755 KIRBY PARKWAY
MEMPHIS, TENNESSEE 38120-4376
901 / 756-6300
FACSIMILE 901 / 757-1296

CHARLES W. METCALF, 1840-1924
WILLIAM P. METCALF, 1872-1940
JOHN W. APPERSON, 1896-1985

OF COUNSEL
JACKSON, SHIELDS,
YEISER & CANTRELL
STEPHANIE GREEN COLE

November 11, 1998

Mr. Geoffrey L. Pratt
Vice President
Cedar Chemical Corporation
24th Floor, Clark Tower
5100 Poplar Avenue
Memphis, TN 38137

Re: W.R. Grace

Dear Geoff:

Enclosed for the company's permanent files is the executed and notarized Release obtained from W.R. Grace & Co. - Conn. in connection with the settlement that was concluded last month. I am closing my file.

Sincerely yours,

Allen T. Malone

ATM:cs
Enclosure

cc: Mr. John C. Bumpers (w/encl.)
Mr. Johnny Hanna (w/encl.)

RELEASE

KNOW ALL MEN BY THESE PRESENTS, that W. R. GRACE & CO.-CONN., a corporation organized and existing pursuant to the laws of the State of Connecticut and having a place of business at 1750 Clint Moore Road, Boca Raton, Florida 33487, as Releasor, in consideration of the sum of One Hundred Five Thousand Dollars ($105,000.00) and other good and valuable consideration, receipt whereof is hereby acknowledged, releases and discharges CEDAR CHEMICAL CORPORATION, a corporation organized and existing pursuant to the laws of the State of Delaware, as Releasee, Releasee's officers, directors, employees, agents, parent, subsidiaries, affiliates, successors and assigns from all actions, causes of action, suits, debts, dues, sums of money, accounts, reckonings, bonds, bills, specialties, covenants, contracts, controversies, agreements, promises, variances, trespasses, damages, judgments, extents, executions, claims, and demands whatsoever, in law, admiralty or equity, which against the Releasee, the Releasor, Releasor's officers, directors, employees, agents, parents, subsidiaries, affiliates, successors and assigns ever had, now have or hereafter can, shall or may have for, upon, or by reason of any matter, cause or thing whatsoever from the beginning of the world to the day of the date of this Release arising from or related to that certain Agreement dated as of March 10, 1989, by and between Cedar Chemical Corporation and W. R. Grace & Co.-Conn.

Whenever the text hereof requires, the use of the singular number shall include the appropriate plural number.

This Release may not be changed orally.

This Release shall be construed, and the performance thereof shall be enforced, in accordance with the laws of the State of New York.

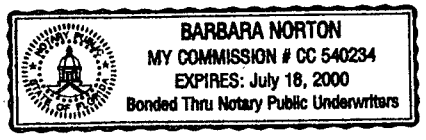
IN WITNESS WHEREOF, the Releasor has caused this Release to be executed by its duly authorized Senior Vice President on October 7th, 1998.

W. R. GRACE & CO.-CONN.

By: W B McGowan
W. Brian McGowan
Senior Vice President

STATE OF FLORIDA)
) ss.:
COUNTY OF PALM BEACH)

On October 7th, 1998, before me personally came W. Brian McGowan, to me known, who, by me duly sworn, did say that he resides in Palm Beach County, Florida, that he is the Senior Vice President of W. R. Grace & Co.-Conn. and that he is authorized to execute this release on behalf thereof.



Barbara Norton
Notary Public

4. Until such time as Grace's Hydrogen Contract with Union Carbide Industrial Gases, Inc., Linde Division, shall have been assigned to Cedar or terminated, Cedar shall reimburse Grace monthly for all such quantities of hydrogen drawn by Cedar for its use and paid for by Grace pursuant to said contract.

5. Grace will make its best efforts to expedite the shipment of all remaining Products and all Raw Materials from the Plant to locations selected by Grace, and both parties will make their best respective efforts to complete the removal and disposal of all wastes generated at the Plant as a result of Cedar's performance under the Agreement, with all such disposal costs for the account of Grace, and all such activities to be completed by not later than the agreed termination date.

6. Grace shall quit claim and relinquish to Cedar any right, title and interest in and to the equipment and fixtures comprising the Plant and all improvements thereto installed in accordance with the provisions of the Agreement.

Please acknowledge the terms of our Agreement set forth above by signing the enclosed duplicate copy of this letter where indicated and returning it to me.

I want to express our appreciation to Grace for the confidence shown in Cedar. We are, of course, sorry that this business has not met Grace's expectations and that Grace has decided to exit the business. If Grace should require contract manufacturing services in the future, we hope that you will call on us again.

Very truly yours,

J. Randal Tomblin

J. Randal Tomblin

JRT:pc

AGREED:
W. R. Grace & Co. - Conn.

By _____
George Power, General Manager

Pratt

CEDAR CHEMICAL CORPORATION

24th Floor • 5100 Poplar Avenue • Memphis, TN 38137 • 901-685-5348

J. Randal Tomblin
Senior Vice President

October 12, 1992

Mr. George Power
General Manager
Organics Chemical Division
W. R. Grace & Co.- Conn.
55 Hayden Avenue
Lexington, MASS 02173

Re: Agreement Between Cedar Chemical Corporation and W. R. Grace & Co. -Conn. Dated 3-10-89

Dear George:

This letter will confirm that you, on behalf of Grace, and I, on behalf of Cedar, have reached agreement on the terms of the early termination of the referenced Agreement requested in your letter to me of September 9, 1992, in accordance with our meeting in Memphis on October 8, 1992.

The provisions of Article 19.2 of the Agreement notwithstanding, we agreed to terminate the Agreement effective December 9, 1992, subject to the following terms and conditions:

1. The termination fee, calculated in accordance with Paragraph 9(c) of the Agreement, in the agreed sum of $1,708,809 (in addition to toll fees invoiced by Cedar to Grace on September 16, 1992 and not yet paid) will be invoiced by Cedar to Grace on or about October 12, 1992, and shall be due and payable by Grace within 30 days of date of invoice.

2. On or about October 15, 1992, Cedar will invoice Grace for the balance of the base fees due under the Agreement for the period September 16, 1992 through December 9, 1992 at the current production fee rate of $150,095 per month. Cedar's invoice for said period of 85 days, in the aggregate amount of $417,504, shall be due and payable 30 days from the date of the invoice.

3. Any amounts invoiced in accordance with the foregoing paragraphs which shall remain unpaid 45 days following the date of such invoices shall bear interest from the due date until date of payment at a rate equal to the prime rate as reported in The Wall Street Journal, plus 2%.

GRACE

Organic Chemicals Division

W. R. Grace & Co.- Conn.
55 Hayden Avenue
Lexington, Mass. 02173

(617) 861-6600

October 23, 1992

Mr. J. Randal Tomblin
Senior Vice President
Cedar Chemical Corporation
2414 Clark Tower
5100 Poplar Ave.
Memphis, Tennessee 78137

Re: Agreement Between Cedar Chemical Corporation and W. R. Grace & Co.-Conn. Dated 3/10/89

Dear Randal:

This letter will confirm that you, on behalf of Cedar, and I, on behalf of Grace, have reached agreement on the terms of the early termination of the referenced Agreement requested in my letter to you of September 9, 1992, in accordance with our meeting in Memphis on October 8, 1992.

The provisions of Article 19.2 and any other provisions of the Agreement notwithstanding, we agreed to terminate the Agreement effective December 9, 1992, subject to the following terms and conditions:

1. The termination fee, calculated in accordance with Paragraph 9(c) of the Agreement, in the agreed sum of $1,708,809 (in addition to $12,096 in toll fees invoiced by Cedar to Grace on September 16, 1992 and not yet paid) will be invoiced by Cedar on or about October 12, 1992.

2. On or about October 15, 1992, Cedar will invoice Grace for the balance of the base fees due under the Agreement, for the period September 16, 1992 through December 9. 1992, at the current production rate of $150,905 per month. To wit, Cedar's invoice for said period of 85 days shall be in the aggregate amount of $427,564.

3. Grace agrees that it is its intent to pay the invoices referenced in Paragraphs 1 and 2 within 45 days of the date of each invoice subject to delays internal to Grace related to processing such payments.

4. Until such time as Grace's Hydrogen Contract with Union Carbide Industrial Gases, Inc., Linde Division, shall have been assigned to Cedar or terminated, Cedar shall reimburse Grace monthly for all such quantities of hydrogen drawn by Cedar for its use and paid for by Grace pursuant to said contract.

5. Grace will make its best efforts to expedite the shipment of all remaining Products and all Raw Materials from the nitroparaffins derivatives plant to locations selected by Grace, and both parties will make their best respective efforts to complete the removal and disposal of all wastes generated at the Plant as a result of Cedar's performance under the Agreement. Cedar shall notify Grace in advance and obtain Grace's approval before engaging in any such removal and disposal activities, all such activities to be completed by not later than the agreed termination date. Grace shall reimburse Cedar for all out of pocket disposal costs which have been approved by Grace.

6. Grace shall quit claim and relinquish to Cedar any right, title and interest in and to the equipment and fixtures comprising the Plant and all improvements thereto installed in accordance with the provisions of the Agreement. Cedar accepts such equipment and fixtures "as is" without any warranties whatsoever from Grace and Cedar accepts full responsibility for the use and operation of such equipment and fixtures.

7. The payment of the sums enumerated above shall constitute full, final and complete payment by Grace for any and all claims by Cedar against Grace under the Agreement.

8. Notwithstanding the termination of the agreement, the provisions of Articles 13 and 14 shall survive termination and continue in full force according to their terms.

Please acknowledge the terms of our Agreement set forth above by signing the enclosed duplicate copy of this letter where indicated and returning it to me.

Very truly yours,

George J. Power
General Manager Nitroparaffins
Organic Chemicals Division

AGREED:

Cedar Chemical Corporation

By: _____
J. Randal Tomblin
Senior Vice President

CEDAR CHEMICAL CORPORATION
P. O. Box 2749, Highway 242S.
West Helena, AR 72390
Phone: (501) 572-3701
Fax: (501) 572-3795

March 30, 1990

Mr. Richard C. Zagraniczny
W. R. Grace & Co.-Conn.
55 Hayden Avenue
Lexington, MA 02173

Re: Procedures for 2-Amino-2-Methyl-1-Propanol Waste Disposal

Cedar Chemical Corporation ("Cedar") agrees to practice the following procedures in the disposal of wastes generated from the manufacture of 2-Amino-2-Methyl-1-Propanol ("AmPro") for W. R. Grace & Co.-Conn. ("Grace").

a) Cedar will complete a waste manifest, a Certificate of Certification and a standard Bill of Lading to accompany each shipment of waste in conformance with all government regulations.

b) Cedar shall ship AmPro aqueous waste to Empak Inc.'s facility in Deer Park, Texas, using transport equipment provided by Trinity Chemical Industries. These will generally be 20,000 gallon railcars, although in the absence of railcars, tank truck shipment is acceptable. Cedar will ensure that the composition of the AmPro aqueous waste shall not exceed the maximum of the range indicated in the attached Generators Waste Profile. Any waste whose composition falls outside this range should not be shipped without prior approval from Grace.

c) Cedar shall ship AmPro aqueous waste containing nickel to Empak Inc.'s facility in Deer Park, Texas, using transport equipment provided by Trinity Chemical Industries. These will be rubber lined tank trucks. Cedar will ensure that the composition of the AmPro aqueous waste containing nickel shall not exceed the maximum of the range indicated in the attached Generators Waste Profile. Any waste whose composition falls outside this range should not be shipped without prior approval from Grace.

d) Cedar shall ship AmPro distillation bottoms to Rollins Environmental Services, Inc.'s facility in Baton Rouge, LA,

using whatever transportation equipment that is appropriate. Cedar will ensure that the composition of the AmPro distillation bottoms shall not exceed the maximum of the range indicated in the attached Generators Waste Profile. Any waste whose composition falls outside this range should not be shipped without prior approval from Grace. Grace may periodically instruct Cedar in writing to ship the AmPro distillation bottoms to an alternate off-site location.

e) Cedar shall ship spent Raney Nickel catalyst to an off-site location designated by Grace for recovery of nickel. Grace shall provide the name of the selected recycler in writing at a later date, as an amendment to this agreement.

f) Cedar shall dispose of all solid wastes other than spent Raney Nickel catalyst that will be generated by the manufacture of AmPro for Grace at a fully permitted Class I facility.

This constitutes the "Procedures" for the disposal of AmPro waste referred to by Articles 4(g) and 13.7 of the March 10, 1989 Agreement between Cedar and Grace for the manufacture of amino alcohols.

Sincerely,

Joe E. Porter
Environmental Engineer

JEP:doc

Attachment

Approvals:

John H. Miles
Cedar Chemical Corporation

3/30/90
Fred Huber
W. R. Grace & Co.-Conn.
Organic Chemicals Division

CEDAR CHEMICAL CORPORATION

24th Floor • 5100 Poplar Avenue • Memphis, TN 38137 • 901-685-5348

April 3, 1990

Mr. Richard Zagraniczny
Product Development Manager
W. R. Grace & Co.
55 Hayden Avenue
Lexington, MA 02173

Dear Mr. Zagraniczny:

As requested, I have signed the four copies of the disposer appendix and have enclosed three, retaining one for Cedar.

Sincerely,

William J. Eissler, Jr. (BD)

William J. Eissler, Jr.
Vice President & General Manager
Organic Chemicals

WJE:bd

Enclosures

GRACE

Organic Chemicals Division
Nitroparaffins Group

W. R. Grace & Co.- Conn.
55 Hayden Avenue
Lexington, MA 02173

(617) 861-6600

April 2, 1990

Mr. William J. Eissler, Jr.
Vice President & General Manager - Organic Chemicals
Cedar Chemical Corporation
24th Floor
5100 Poplar Avenue
Memphis, TN 38137

Dear Mr. Eissler:

It appears that Empak requested a wording change in the disposer appendix (marked in red). Please resign the four enclosed originals, keep one for Cedar's files and forward the remaining three to me.

Sincerely,

Richard C. Zagraniczny
Product Development Manager

RCZ:doc

Enclosures

Telex: 200076 GRLX UR
FAX: 617-863-8070
TWX: 710-326-0744

DISPOSER APPENDIX

EMPAK INC., hereinafter referred to as Contractor, represents and warrants that it understands the currently known hazards which are presented to persons, property and the environment in the disposal of the wastewaters listed in the Generators Waste Profiles, hereinafter referred to as Material, and acknowledges that W. R. Grace & Co. - Conn., hereinafter referred to as WRG, and its toll manufacturer Cedar Chemical Corporation, referred to as CCC, relies on said representations and warranties; that it will dispose of the Material in full compliance with all governmental laws, regulations and orders; and that the Disposal Facility above described is now licensed and permitted pursuant to all local, state and federal laws and regulations, to accept and dispose of the Material.

Except in the case of negligence, willful falsification of documents, or willful misconduct on the part of WRG or CCC, their employees, agents, or representatives, and except with respect to Material not conforming to the description of Material set forth in appropriate Attachments, upon delivery and acceptance by Contractor of WRG's Material, WRG and CCC will be relieved from any further obligation with regard to disposal of the Material, and Contractor shall indemnify and hold harmless from and against (1) any fine or penalty; and (2) any loss, damage, suits, liability, and expenses (including, but not limited to, reasonable investigation and legal expenses) arising out of any claim for loss of or damage to property, including WRG's and CCC's employees, which may reasonably be incurred by or imposed upon WRG or CCC as a result of Contractor's failure to store, treat, process, or dispose of the Material in accordance with the provisions of this Agreement.

The indemnity and hold harmless obligations outlined in the immediately preceding section hereof shall apply reciprocally from WRG to Contractor for any claim for sudden and accidental spills and contamination (including fine or penalty) or any loss of or damage to property and injuries to or death of persons which result from (1) WRG's or CCC's negligence or willful misconduct in the transfer of the Material to Contractor in accordance with the provisions of this Agreement and/or (2) WRG's or CCC's failure to maintain its storage and transferring equipment in accordance with the provisions of this Agreement. WRG and CCC also shall indemnify, reimburse, and hold Contractor harmless for any losses, damages, penalties, fines, or civil liabilities incurred by Contractor as a result of the nature of any Material not conforming to the descriptions of Material in Attachments hereto, including, but not limited to, losses associated with the delivery, storage, cleanup, treatment, or disposal of nonconforming Material.

In the event any claim or action shall be made or brought against an indemnified party in respect of which indemnity may be sought against the indemnifying party pursuant to the foregoing provisions, the party shall promptly notify the indemnifying party in writing and the indemnifying party shall assume the defense thereof, including the employment and payment of counsel.

IN WITNESS WHEREOF, the parties hereto have duly executed this Agreement of March 1st, 1990.

EMPAK INC.

Signature: David L. Glover

Name: DAVID L. GLOVER

Title: Vice President

W. R. GRACE & CO. - CONN.
Organic Chemicals Division

Signature: [signature] 3/30/90

Name: FRED HUBER

Title: Exec. V.P.

TRINITY CHEMICAL INDUSTRIES, INC.

Signature: [signature]

Name: TERRY L FISHER

Title: Vice-PRESIDENT

CEDAR CHEMICAL CORPORATION

Signature: [signature]

Name: WILLIAM J. EISSLER, JR

Title: VP- Organic Chemicals

CEDAR CHEMICAL CORPORATION
P. O. Box 2749, Highway 242S.
West Helena, AR 72390
Phone: (501) 572-3701
Fax: (501) 572-3795

March 7, 1990

Mr. Richard C. Zagraniczny
W. R. Grace & Co.-Conn.
55 Hayden Avenue
Lexington, MA 02173

Re: Procedures for 2-Amino-2-Methyl-1-Propanol Waste Disposal

Cedar Chemical Corporation ("Cedar") agrees to practice the following procedures in the disposal of wastes generated from the manufacture of 2-Amino-2-Methyl-1-Propanol ("AmPro") for W. R. Grace & Co.-Conn. ("Grace").

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c) Cedar shall ship AmPro aqueous waste containing nickel to Empak Inc.'s facility in Deer Park, Texas, using transport equipment provided by Trinity Chemical Industries. These will be rubber lined tank trucks. Cedar will ensure that the composition of the AmPro aqueous waste containing nickel shall fall within the range indicated in the attached Generators Waste Profile. Any waste whose composition falls outside this range should not be shipped without prior approval from Grace.

d) Cedar shall ship AmPro distillation bottoms to Rollins Environmental Services, Inc.'s facility in Baton Rouge, LA,

using whatever transportation equipment that is appropriate. Cedar will ensure that the composition of the AmPro distillation bottoms shall fall within the range indicated in the attached Generators Waste Profile. Any waste whose composition falls outside this range should not be shipped without prior approval from Grace. Grace may periodically instruct Cedar in writing to ship the AmPro distillation bottoms to an alternate off-site location.

e) Cedar shall ship spent Raney Nickel catalyst to an off-site location designated by Grace for recovery of nickel. Grace shall provide the name of the selected recycler in writing at a later date, as an amendment to this agreement.

f) Cedar shall dispose of all solid wastes other than spent Raney Nickel catalyst that will be generated by the manufacture of AmPro for Grace at a fully permitted Class I facility.

This constitutes the "Procedures" for the disposal of AmPro waste referred to by Articles 4(g) and 13.7 of the March 10, 1989 Agreement between Cedar and Grace for the manufacture of amino alcohols.

Sincerely,

Joe E. Porter
Environmental Engineer

JEP:doc

Attachment

Approvals:

Geoffrey L. Pratt
Cedar Chemical Corporation

Peter I. Kiziuk
W. R. Grace & Co.-Conn.

JUL 5 1989

CEDAR CHEMICAL CORPORATION

24th Floor • 5100 Poplar Avenue • Memphis, TN 38137 • 901-685-5348

June 29, 1989

Mr. R. C. Zagraniczny
Product Development Manager
Nitro Paraffins Group
W. R. Grace & Co. - Conn.
55 Hayden Avenue
Lexington, Massachusetts 02173

Dear Mr. Zagraniczny:

This letter will confirm the Agreement which you reached with Geoff Pratt, whereby, throughout the term of the Hydrogen Supply Agreement between Union Carbide (Linde), Grace and Cedar, Cedar shall be entitled to draw hydrogen from the tank to be supplied by Linde at such times as Cedar shall require hydrogen for use in connection with projects other than the Grace project. Such hydrogen, when so consumed, shall be separately metered and Cedar will advise Grace of such consumption at the end of each month. Since the cost of any hydrogen used by Cedar for its own account will be billed by Linde to Grace in accordance with the above mentioned Agreement, Cedar will reimburse Grace its actual cost for such hydrogen, based on the pricing determined by the Agreement. Reimbursements will be made upon demand by Grace, accompanied by documentation of the unit cost paid by Grace to Linde. It is also understood that at no time will Cedar draw such quantities of hydrogen as would interefere with Cedar's ability to perform in accordance with its Agreement with Grace dated March 10, 1989.

Please acknowledge the foregoing by signing and returning the enclosed copy of this letter.

Sincerely yours,

William J. Eissler, Jr.
Vice President and General Manager
Organic Chemicals

WJE:nm
Enclosure

AGREED:
W. R. GRACE & CO. CONN.

By: Edward G. Najjar

Organic Chemical Division
Nitroparaffins Group

GRACE

W.R. Grace & Co.
55 Hayden Avenue
Lexington, Mass. 02173

(617) 861-6600

March 15, 1989

Mr. Geoffrey L. Pratt
Director of Operations, Custom Manufacturing
Cedar Chemical Corporation
24th Floor
5100 Poplar Avenue
Memphis, Tennessee 38137

Dear Geoff:

First of all, I would like to say that I and everyone at the Organic Chemicals Division are just delighted to hear that Cedar Chemical has signed the contract. We look forward to a long and mutually profitable relationship.

Regarding hydrogen supply and your new project, we have no problems with sharing the liquid hydrogen handling system where possible. I propose we deal with it as follows:

a) The variable portion of the cost or per pound hydrogen price is negotiated by Grace using the combined Grace and Cedar volume requirements. The final per pound price is the same for Cedar and Grace.

b) The fixed charge or equipment rental charge to be shared by each party based on each party's percent of forecast hydrogen consumption. Reconciliation based on metered consumption to be determined at the end of each quarter.

Please signify that the arrangement is acceptable by signing below. You should let me know your anticipated 1990 consumption, the time you intend to start consumption, and your forecast for 1991.

Sincerely,

Richard C Zagranczny

Richard C. Zagraniczny
Product Development Manager

RCZ:doc

Approved by: William Cirila Jr
Title: Vice Pres & Gen Mgr
Date: 3-31-89

Telex: 200076 GRLX UR
FAX: 617-863-8070
TWX: 710-326-0744

To John Mills

Copy

AGREEMENT

THIS AGREEMENT made as of the 10th day of MARCH, 1989, by and between **Cedar Chemical Corporation**, a Delaware corporation with offices at Suite 2414, Clark Tower, 5100 Poplar Avenue, Memphis, Tennessee 38137 ("Cedar") and **W. R. Grace & Co.-Conn.**, a Connecticut corporation with offices at 55 Hayden Avenue, Lexington, Massachusetts 02173 ("Grace").

WHEREAS, Grace has processes and technology for the production of aminoalcohols from nitroparaffins; and

WHEREAS, Cedar owns production facilities located at West Helena, Arkansas, which when modified in the accordance with the provisions of this Agreement, are deemed by Grace to be capable of manufacturing aminoalcohols from nitroparaffins to be supplied by Grace in accordance with the provisions hereof; and

WHEREAS, Grace desires to retain Cedar, acting as independent contractor, to produce aminoalcohols and other products for it during the term of this Agreement; and

WHEREAS, Cedar is willing to construct modifications of its said manufacturing facilities and to produce products for Grace in accordance with processes and process engineering approved by Grace, using raw materials supplied by Grace, all in accordance with the provisions hereof.

NOW, THEREFORE, in consideration of the premises and the mutual covenants contained herein, the parties agree as follows:

1. Definitions: For purposes of this Agreement the following terms shall have the meanings assigned thereto:

1.1. **Plans** shall mean those detailed plans and specifications attached hereto as Exhibit A or referred to in said exhibit detailing the Plant which Cedar shall construct.

1.2. **Plant** shall mean that portion of Cedar's West Helena facilities identified in Exhibit B, including the modifications described in the Plans, for the production of the Products.

1.3. **Product or Products** shall mean those products described and meeting the specifications contained in Exhibits C-1 through C-3 inclusive, as well as such additional products as the parties shall identify in supplemental exhibits to be attached hereto.

1.4. **Specifications** shall mean the Product specifications contained in Exhibit C-1 through C-3 as they may be modified or added to from time to time by mutual agreement of the parties as provided herein.

1.5. **Process or Processes** shall mean those processes supplied by Grace to Cedar hereunder identified in Exhibits D-1 through D-3, as well as any supplemental processes identified in supplemental exhibits to be attached hereto, which Grace deems adequate to produce the corresponding Products identified in Exhibit C-1 through Exhibit C-3 and supplements thereto.

1.6. **Raw Materials** shall mean those nitroparaffins and other raw materials meeting the specifications contained in Exhibits E-1 through E-3 as well as any supplemental raw materials as shall be attached hereto, as shall be required for the productions of Products, including all necessary containers and shipping and packaging materials as shall be required for Cedar to perform in accordance with the provisions of this Agreement.

1.7. **Plant Start-Up** shall mean the period beginning with the first introduction of Raw Materials into the Plant for the purpose of producing Product for Grace hereunder until the earlier of (i) 2,000 pounds of Product meeting Specifications are produced in an eight hour time period or (ii) Grace notifies Cedar that Plant Start-Up is deemed complete.

1.8. **Product Start-Up** shall mean the period of the first attempt at commercial production of each Product in the Plant, as provided in Section 5.2 of this Agreement.

1.9. **Contract Year** shall mean each successive twelve (12) month period which commences on the first day of the month next following the beginning of Plant Start-Up; provided that the fifth Contract Year shall be extended an additional term equal to the period between the first day of the first Contract Year and the completion of Plant Start-Up.

1.10. **Effective Date** shall mean the date first appearing hereinabove.

2. Term:

This Agreement shall commence on the date hereof and shall continue for a term ending on the last day of the fifth Contract Year (hereinafter the "Initial Term"). Grace shall have the right in its sole discretion to extend the term of this Agreement for one (1) additional period of up to five (5) years (the "Extended Term") upon the same terms and conditions hereunder at the time of extention, MUTATIS MUTANDIS, except as follows:

(a) The Toll Fee set out in Article 9(b) shall not exceed 18¢ per pound increased by the percentage change in the Producer Price Index as published by the Department of Labor (or any replacement index) from the Effective Date to the effective date of Grace's extension of the Agreement, plus 10¢; and

(b) Grace shall take or pay for (at the Toll Fee stated hereinabove) a minimum of 3,000,000 pounds of Product from Cedar during each Contract Year during the Extended Term.

(c) Grace shall give written notice to Cedar of any such extension at least one hundred eighty (180) days prior to the expiration of the Initial Term.

(d) The parties understand that any Plant rehabilitation reasonably required to permit Cedar to perform hereunder during the Extended Term shall be for Grace's

account. For purposes of this paragraph, Plant rehabilitation shall mean replacement or overhaul of any major item of equipment included in the Plant, at a cost of more than $10,000, but shall not include repairs and maintenance by Cedar hereunder in the ordinary course of business, consistent with the scope of Cedar's work hereunder.

3. Plant Modifications And Start-Up:

3.1. Cedar shall modify the Plant in accordance with the Plans attached hereto as Exhibit A, which Plans have been approved by Grace and which, when implemented by Cedar in accordance with good construction and engineering procedures are deemed by Grace adequate to permit the production of Products hereunder at rates up to 4,000,000 pounds per year. Grace shall provide qualified on-site personnel to consult with Cedar in such modification, it being agreed, however, that the construction of the Plant shall be the sole responsibility of Cedar. Cedar shall make its best efforts to complete the modifications within nine (9) months following the Effective Date. Title to all equipment and other improvements purchased and installed in accordance with the Plans and title to the Plant shall vest and remain solely in Cedar, subject to Grace's right to cause Products to be produced for it by Cedar in the Plant, in accordance with the terms hereof, during the entire term of this Agreement.

3.2. It is understood and agreed that Cedar's cost of implementing the Plans shall be recovered by Cedar in accordance with the provisions of Articles 9(b) and (c). In the event Grace requests that Cedar implement other or additional modifications not identified in Exhibit A, Cedar shall do so only if the cost thereof shall be borne by Grace, such cost to be billed to Grace as incurred by Cedar and due and payable by Grace thirty (30) days thereafter.

3.3. It is understood and agreed that the sufficiency of the Processes to enable Cedar to produce Products in the Plant is Grace's responsibility. In the event the Plant fails to perform as contemplated herein, Cedar will will make its best efforts to assist Grace to develop modifications in the Plans and/or Processes in order to achieve Grace's objectives hereunder, provided that the costs associated with any such modifications not reflected in the exhibits attached hereto shall be borne by Grace. If the Plant fails to perform as contemplated herein as a result of Cedar's failure to adhere to the Plans, or faulty construction of the Plant (including purchase or use of defective equipment in connection therewith) or Cedar's failure to perform its obligations set out in Article 4 hereof, including failure to adhere to the Processes, then the cost of any required modifications to remedy such failure shall be borne solely by Cedar.

4. Scope of Work:

In each Contract Year during the term hereof, Cedar shall perform the following services for Grace:

(a) Reserve the entire capacity of the Plant for the manufacture of Products for Grace;

(b) Provide labor facilities, utilities, and support services for the Plant at no additional charge to Grace, and provide nitrogen to the Plant for a charge equal to Cedar's cost plus 5%, as shall be necessary and appropriate to enable Cedar to manufacture Products for Grace.

(c) Maintain the Plant in a condition consistent with the quality requirements of the Products; the maintenance of good control of operating conditions; the safety of Cedar's Plant employees; the minimization of Raw Material losses; the requirements of all applicable laws and regulations concerning the manufacture of Products; and good maintenance practices.

(d) Make its best efforts to manufacture and deliver to Grace from Raw Materials supplied by Grace such quantities of Products as Grace shall order hereunder, meeting the Specifications.

(e) Adopt reasonable raw material usage standards based on Product yields achieved during the initial production campaign, to be adjusted for yields

achieved in subsequent production campaigns, for each Product to be manufactured hereunder, and to reimburse Grace for any losses incurred as a result of the failure to achieve such standards in connection with subsequent production campaigns.

(f) Prepare or package the Products for shipment in accordance with Grace's shipping instructions, using packaging (bulk sacks or 55 gallon drums) and shipping materials supplied by Grace.

(g) Cedar shall arrange for disposal of all wastes generated by or from its manufacture of Products and/or its use of the Process hereunder, using properly licensed transporters and off-site, third-party disposal facilities and will invoice Grace, or arrange for Grace to be invoiced for the actual costs so incurred. Cedar shall submit in writing and Grace shall approve in advance Cedar's procedures for the disposal of all such waste (the "Procedures"). Cedar shall make its best efforts to develop methods for disposing of wastes on the Plant site in accordance with such permit requirements and governmental regulations as shall be applicable thereto, it being understood that the parties shall share any cost savings realized as a result of on-site waste disposal undertaken by Cedar hereunder.

5. **Manufacturing Schedules:**

5.1. During the term of this Agreement, beginning as of the first day of the first Contract Year and continuing monthly thereafter, Grace shall notify Cedar whether the next succeeding month shall be designated a Production Month or an Idle Month. For the purposes of this Agreement, an Idle Month is defined as a month in which Cedar will provide all normal services within the scope of work specified in Article 4 herein other than actual production of Product. A Production Month shall be any calendar month in which Grace shall have directed Cedar to produce Product hereunder.

5.2. For purposes of this Agreement, the period of the first production campaign for each individual Product shall be designated a Product Start-Up period. During a month in which Product Start-Up is occurring, Cedar shall assign the majority of its technical staff at its West Helena Facility to the Plant to facilitate the start-up operation. Grace shall determine in its discretion when Product Start-Up is completed and when Grace so determines, the Plant shall revert to Production Month Status.

5.3. In no event shall the duration of any campaign for production of Products ordered by Grace hereunder be less than thirty (30) days, nor shall Cedar be required to initiate more than two separate production campaigns for any one Product in any Contract Year without Cedar's prior consent.

5.4. Standards for Raw Material usage and Product throughput will be determined for each individual Product at the completion of Product Start-Up. If through no fault of Cedar Product meeting the Specification is not produced during Product Start-Up, the parties shall adopt revised Specifications for such Product based on results achieved during such Product Start-Up. Any improvement in Product Specifications, Raw Material usage, and Product throughput will be determined by Cedar and Grace after each subsequent production campaign, whereupon new standards for said factors shall be adopted based on performance during such campaign. Any Raw Material usage less favorable than such standards shall be at Cedar's cost and expense, while any economic benefit from Raw Material usage more favorable than such standards shall be shared equally by Grace and Cedar.

5.5. The parties shall include additional Products among those to be manufactured by Cedar hereunder at such times as Grace shall have disclosed to Cedar the results of pilot test production of such Products and submitted appropriate supplements to Exhibits C, D, E, F and G provided that if the manufacture of such additional Products would require Cedar to materially increase the number of employees committed to such manufacture or increase the level of risk associated with such manufacture, or affect any material reduction in the rate of production of Products, then Cedar

and Grace shall negotiate an appropriate modification to the Toll Fees and Base Fees hereunder.

5.6. Grace will provide Cedar a calendar quarterly production forecast in writing thirty (30) days before the beginning of each calendar quarter. It is recognized by the parties that such notices are for planning purposes only.

6. **Title and Risk of Loss:**

Title to all Raw Materials supplied to Cedar by Grace, and title to all Products manufactured therefrom shall at all times remain in Grace. Risk of loss of Raw Materials while in Cedar's custody, possession or control, whether in their original state as supplied by Grace, or after conversion to Product shall be the responsibility of Cedar. Cedar shall provide property insurance covering all direct risks of loss to Products and Raw Materials owned by Grace while in Cedar's possession in amounts not less than Grace's cost. Cedar shall cause the insurance policy providing such coverage to be endorsed to name Grace as an additional insured thereunder, as its interest shall appear.

7. **Quality Control:**

7.1. Grace will provide or cause Cedar to be provided a weight ticket and a certificate of analysis for all Raw Materials which it delivers or causes to be delivered to Cedar hereunder, certifying such Raw Materials to be in

accordance with the specifications identified in Collective Exhibit E.

7.2. Cedar shall inspect all Raw Materials tendered by or on behalf of Grace and shall promptly advise Grace of any defects in such Raw Materials, using the applicable methods of analysis set forth in Collective Exhibit F.

7.3. Cedar shall sample and analyze each batch of Products produced by it to determine whether they meet the applicable Specifications, using the methods of analysis set forth in Collective Exhibit G.

7.4. Cedar shall retain product samples and records of analyses performed in accordance with this Article 7 for a period of one (1) year following production. Prior to Cedar's disposal of such samples and records, Cedar shall offer the samples and records to Grace. At Grace's request, Cedar shall prepare and ship samples of materials and Products to Grace with shipping costs for Grace's account.

8. Minimum Purchase Order Requirements:

During the Initial Term, Grace shall make its best efforts to issue purchase orders to Cedar for production of Products, and to deliver Raw Materials to Cedar in sufficient quantities to enable Cedar to produce such Products, in the following minimum quantities (dry pound basis):

First Contract Year - 2,000,000 pounds

Second Contract Year - 2,500,000 pounds

Third Contract Year - 3,000,000 pounds

Fourth Contract Year - 3,500,000 pounds

Fifth Contract Year - 4,000,000 pounds

Cumulative Total
For Initial Term: - 15,000,000 pounds

In the event Grace shall not order its total requirements for Products in any Contract Year during the term hereof, Cedar shall have the right to use the Plant for production of materials other than Products for its own account, following written notice to and approval by Grace, which approval shall not be unreasonably withheld; provided, however, the Base Fees which otherwise would be due hereunder during any such period of Cedar's use of the Plant for its own account shall be waived, and all quantities of materials so produced by Cedar shall be credited against Grace's minimum Product purchase obligations and shall also be credited as Product produced in determining any termination payment due pursuant to Article 9(c). Cedar's use of the Plant shall not interfere with Cedar's production obligations for Products.

9. **Fees**:

As compensation for the services rendered, expenses incurred, facilities provided and obligations assumed by Cedar hereunder, Grace shall pay Cedar the following fees during the term hereof:

(a) Base Fees - Beginning thirty (30) days following initiation of Plant Start-Up, and monthly thereafter, Grace shall pay to Cedar the sum of One

Hundred Seventy Five Thousand Dollars ($175,000) with respect to each such preceding month that shall be designated under Article 5 a Product Start-Up Month; the sum of One Hundred Forty Thousand Dollars ($140,000) with respect to each such preceding month that shall be designated under Article 5 a Production Month, and the sum of One Hundred Thousand Dollars ($100,000) for each such preceding month that shall be designated under Article 5 an Idle Month. Said monthly base fees shall be prorated proportionately for any month which is a combination of an Idle Month/Production Month/Product Start-Up Month. Grace shall be relieved of its obligation to pay the Base Fees during the term hereof only for any period in which Cedar is unable to produce Product as contemplated hereunder as a result of Cedar's failure to carry out its obligations hereunder or as a result of labor disputes or a strike by Cedar's employees, or by any employees engaged to carry out the Plant modifications pursuant to Article 3, or a result of the partial or total destruction of the Plant by fire, explosion or other insurable casualty which prevents Cedar's ability to produce Product ordered by Grace for more than thirty (30) days following such event.

(b) Toll Fees - Grace shall pay Cedar a Toll Fee in the sum of 38¢ per pound FOB the Plant for all Product produced for Grace since the Effective Date

until the quantity produced during each Contract Year, when added to the quantity produced since the Effective Date, shall exceed the aggregate minimum quantity of Products to be ordered by Grace as of the end of such Contract Year as specified in Article 8, whereupon the fee for all quantities of Product in excess of such aggregate minimum produced by Cedar during the remainder of such Contract Year shall be reduced by the sum of 20¢ per pound. It is recognized that, for purposes of this Article 9(b), Toll Fees will be due with respect to all Products produced for Grace during each Product Start-Up period, whether or not such Product meets the Specifications, unless failure of Product to meet such Specifications shall have been caused by Cedar's default in its obligations hereunder. In the event the Product Specifications are not achieved during such Product Start-Up, Product produced following conclusion of such Product Start-Up shall be required to meet those Specifications achieved during Product Start-Up as shall be accepted by Grace pursuant to the provisions of Article 5.4.

(c) Minimum Annual Toll Fees - It is further agreed that if for any reason during the term of this Agreement Grace shall order and Cedar shall produce less than the minimum quantity of Product specified in Article 8 with respect to any Contract Year, Grace shall pay to Cedar, in addition to all other fees due

hereunder, a sum equal to the difference between such minimum quantity of Product and the number of pounds of Product actually ordered by Grace and produced by Cedar during such Contract Year times 20¢; provided that, to the extent Grace shall pay minimum Toll Fees hereunder with respect to any quantity of Product ordered by Grace which Cedar shall not have produced due to any reason specified in the last sentence of Article 9(a), such fees shall be credited to the Toll Fees payable by Grace for Product to be purchased by Grace hereunder, amortized over the remaining term of the Agreement. Quantities of Product that were produced and paid for before the first day of the first Contract Year shall be included in determining any minimum annual Toll Fee for the first Contract Year. In the event this Agreement shall terminate at any time prior to the expiration of the Initial Term, for any reason other than (1) Cedar's repudiation of its obligations hereunder or (2) for any other reason justifying termination by Grace in accordance with the provisions of Articles 19.3 or 19.4, (in which event no payment shall be due by Grace) Grace shall pay to Cedar, in addition to all fees and costs otherwise due as of the date of such termination, a sum equal to $3,000,000 less credit for Product produced and invoiced by Cedar (or otherwise paid for by Grace or credited to Grace pursuant to the first sentence of this

Article 9(c)) hereunder at the rate of 20¢ per pound, which sum, if paid by Grace within ninety (90) days following termination, shall be accepted by Cedar in full and final settlement and satisfaction of all claims against Grace arising out of the early termination of this Agreement.

(d) Fee Escalation - The Base Fees identified in Article 9(a) shall be subject to escalation no more frequently that once every twelve (12) months, but in no event effective earlier than the first day of the Second Contract Year. Such fee escalations shall be established by written notice to Grace at least thirty (30) days prior to the effective date thereof, and shall be determined in accordance with the following formula:

.53 x (% increase in average hourly rate for Plant employees) +

.06 x (% increase in electric rate) +

.03 x (% increase in the gas rate) +

.38 x (% increase in the consumer price index)

The first increase shall be based on changes from the first day of the first Contract Year (the Base Period); thereafter increases shall be based on changes from the Base Period to the effective date of each escalation. All percentage increases shall be applied against original Base Fees identified in Article 9(a). Cedar's noti-

ces to Grace of all increases shall be accompanied with reasonably adequate documentation with respect to each of the escalation factors stated hereinabove.

10. Billing and Payment Schedule:

Cedar shall submit itemized invoices to Grace as of the first day of each month during each Contract Year during the term hereof covering the Base Fee with respect to the previous month and the Toll Fee with respect to Product produced during the previous month. Any minimum Toll Fee, if applicable, shall be invoiced as of the last day of each Contract Year. All such invoices shall be due and payable within thirty (30) days of the latter of the date of invoice or the date of mailing of such invoice.

11. Access to the Plant/Assistance:

11.1. Upon reasonable notice, Grace and its representatives shall have access to the Plant at all times during normal working hours. Grace agrees, subject to the provisions of Article 13.6, to defend, indemnify and hold Cedar harmless against any and all claims and causes of action asserted against Cedar on account of personal injury or property damage sustained by Grace personnel or representatives while present at Cedar's Plant, except as shall have been caused by the negligent act or omission of Cedar, its agents or representatives.

11.2. During the period of Cedar's modification of the Plant and during each Product Start-Up period, Grace shall provide Cedar with such on-site personnel as shall be reasonably necessary to assist Cedar in completion of the said modifications and Plant Start-Up and for each Product Start-Up.

12. **Warranties:**

Cedar warrants that each Product produced by it hereunder following the conclusion of such Product Start-Up shall be produced in accordance with the provisions of the Agreement and shall meet the Specifications, or such revised Specifications as the parties shall mutually adopt in writing in accordance with Article 5.4, and further, that such Product shall be delivered to Grace free of any liens or encumbrances. Cedar makes no other warranty with respect to the Products to be manufactured hereunder whether of merchantability or fitness for a particular purpose and none shall be implied.

13. **Indemnification:**

13.1. Cedar acknowledges that hazards may be involved in providing the services described hereunder and represents that it is knowledgeable and capable of providing such services in a professional and safe manner. Cedar shall take all necessary and reasonable precautions in its processing, handling, transportation and disposal of Raw

Materials and Products. Grace may provide Cedar with certain information regarding the Raw Materials and Products, including procedures for processing, handling and disposal as well as toxicological data. Such information is provided for informational purposes only and without any representation as to its completeness or suitability in providing the services described herein. The methods employed and the precautions taken to handle and use Raw Materials and Products shall be determined solely by Cedar.

13.2. Cedar shall indemnify, defend and hold Grace harmless from and against any and all liability, losses, expenses, interest, claims, demands, causes of actions, damages, costs and reasonable attorneys fees based upon or arising out of any injury, illness and/or death of any person including Cedar's employees, or damage to or destruction of any property arising out of or in connection with the work performed by Cedar hereunder, except to the extent that any such claim or liability arises out of the breach by Grace of the terms and conditions of this Agreement or its obligations and warranties hereunder.

13.3. Grace shall indemnify, defend and hold Cedar harmless from and against any and all liability, losses, expenses, interest, claims, demands, causes of action, damages, costs and reasonable attorneys fees based upon or arising out of any injury, illness and/or death of any person or damage to or destruction of any property incident to the shipment of Raw Materials to Cedar hereunder or the sale,

use, shipment, handling or other disposition of Products manufactured hereunder following delivery of same by Cedar to Grace, except to the extent caused by Cedar's breach of its obligations or warranties hereunder.

13.4. Cedar agrees to carry the following minimum insurance coverage during the term hereof:

(a) Workers Compensation and Employers Liability Insurance in an amount sufficient by virtue of the laws of the State of Arkansas; and

(b) General Public Liability Insurance in an amount equal to $1,000,000 per occurrence and $5,000,000 annual aggregate; and

(c) Contractual Liability Insurance to cover the liability herein assumed by Cedar with limits of liability not less than those stated above.

All of the above insurance policies shall name Grace as an additional insured party as its interest shall appear and shall contain a clause prohibiting cancellation except upon thirty (30) days prior written notice to Grace.

13.5. Grace warrants that no Process, when used to manufacture Product hereunder, will, in and of itself, infringe any valid United States patent, and, subject to the provisions of Article 13.6, Grace shall indemnify and hold Cedar harmless from costs and damages, including reasonable

attorneys fees incurred by Cedar as a result of any such patent infringement claim. If in Grace's opinion, the continued use of the Processes to manufacture Products would constitute patent infringement, Grace may terminate this Agreement upon notice in accordance with Article 19.2, and subject to the provisions of Article 9(c), without further liability to Cedar for lost profits, lost opportunities, or other consequential damages to Cedar's business. It is further understood that Grace makes no warranty against patent infringement and shall have no obligation hereunder as a result of (i) any changes in the Processes made by Cedar without Grace's prior written approval, (ii) the use by Cedar of any technical data, information or manufacturing process not encompassed within the Processes, (iii) the use by Cedar of any equipment, machinery or processes used in Cedar's discretion to make the Products.

13.6. The provisions of this Article 13 with respect to indeminification shall not apply or be effective with regard to any claim, demand, suit or action (other liabilities, losses, damages, costs or expenses relating thereto or arising therefrom) unless:

(a) The indemnitee has advised the indemnitor promptly in writing of any such claim, demand, suit or action;

(b) The indemnitee shall give the indemnitor all reasonable cooperation and assistance in the defense of any such claim, demand, suit or action;

(c) The indemnitee shall afford the indemnitor the unqualified opportunity of directing the defense of any such claim, demand, suit or action at indemnitor's discretion and expense with counsel selected by indemnitor, and

(d) The indemnitee shall refrain from compromising or settling any such claim, demand, suit or action or seeking to do so without indemnitor's prior consent in writing, which consent may be withheld by indemnitor in its discretion.

The settlement of any claim, demand, suit or action without the indemnitor's prior written consent to the terms and conditions of such settlment shall discharge any obligation the indemnitor might otherwise have under this Article 13, but only in respect of such specific claim, demand, suit or action or any liability arising directly therefrom.

13.7. Cedar warrants that it will arrange for disposal of all wastes in strict conformance with the Procedures referred to in Article 4(g) and that it will not modify the Procedures, including but not limited to changing

the transporter or the disposal site for such wastes, without first notifying Grace and obtaining Grace's written approval, and Cedar further warrants that, to the extent that Cedar is directly involved in such waste disposal activities, it will be in compliance with all applicable governmental laws, rules, regulations and orders. Cedar shall indemnify and hold Grace harmless from and against all liability arising from Cedar's breach of the above-stated warranty. Grace shall indemnify and hold Cedar harmless from and against all liability arising from disposal of such waste, except if and to the extent due in whole or in part to Cedar's breach of the above stated warranty.

13.8. Notwithstanding anything to the contrary contained herein, the provisions of the Article 13 shall remain operative and in full force and effect regardless of:

(a) The consummation of the sale and purchase of any Product, and

(b) Any investigation made by or on behalf of the indemnitee.

14. **Proprietary Information:**

The Secrecy Agreement dated September 16, 1987, between Cedar and Grace with respect to the Products and Processes referred to herein, including any additional Products and Processes which shall be identified following the Effective Date hereof and exhibited hereto as supplemen-

tal exhibits, is incorporated herein by reference and shall continue in full force and effect.

15. **Compliance With Law and Government Regulations:**

15.1. Cedar shall store and handle all Raw Materials and Products in accordance with generally accepted safety standards. In the conduct of its operations hereunder, Cedar shall comply with all federal, state and local laws, regulations, and ordinances applicable to its scope of work hereunder; provided that in the event any statute, regulation, or governmental order enacted or promulgated subsequent to the effective date of this Agreement shall impose additional compliance costs on Cedar directly related to the Products to be manufactured hereunder, Cedar shall promptly notify Grace of such additional compliance costs that would be required in order to satisfy such provision. Upon being informed of such costs, Grace shall have the right either to pay such incremental costs or terminate this Agreement in accordance with Article 19.

15.2. Grace warrants to Cedar that to the extent applicable to Grace in respect of any Product to be manufactured by Cedar hereunder, it has or will at the appropriate time comply fully with the provisions of the Toxic Substance Control Act.

16. **Force Majeure:**

Subject to the provisions of Article 19, the performance by either party of any obligation on its part to be

performed hereunder (other than an obligation to pay money) shall be excused if such performance is prevented by act of God or the public enemy, fire, explosion, flood, drought, epidemic, quarantine, restrictions, war, insurrection, riot, sabotage, embargo, strikes, accidents, injunctions or restraints of govenrment, compliance with any order or regulations of Federal or State governments or agencies thereof, shortage of materials or energy, or any other cause beyond the reasonable control of the party failing to perform, provided, however, that the party affected shall provide written notification to the other party setting forth the grounds for its nonperformance and shall exert its best efforts to eliminate or cure or overcome any of such causes to resume performance of its obligations hereunder. Any period during which either party's non-performance is excused hereunder shall be added to the term of the fifth Contract Year (or, during an Extended Term, such period shall be added to the final Contract Year during such Extended Term) so that the full term of the Agreement shall be given effect unless this Agreement has been terminated as otherwise provided in this Agreement.

17. **Material and Product Accounting**:

17.1. Cedar shall account for all Raw Materials and Products by maintaining a complete set of records showing in detail the quantities of Grace-owned Raw Materials received,

processed, balance on hand, waste/loss and shipped, as well as any other detail requested by Grace. Cedar shall also submit separate monthly written reports for each material to Grace.

17.2. Allowance shall be reconciled quarterly, net of gains, within thirty (30) days of the end of the quarter, and shall not be carried over to subsequent quarters.

17.3. Cedar shall permit Grace's duly authorized representatives to audit its books and records and to verify the entries through physical audits conducted during Cedar's normal business hours. A physical inventory will be taken and an inventory reconciliation will be made at least quarterly with personnel from both Cedar and Grace present and at such other times as either Grace or Cedar elects for all Raw Materials and Products. Cedar's cost for the quarterly physical inventory and inventory reconciliation will be absorbed by Cedar. More frequent physical inventories and reconciliations not to exceed once per calendar month may be conducted at the expense of the inspecting party.

18. **Notices**:

18.1. All notices hereunder shall be deemed to be properly served or sent if by registered mail with postage prepaid thereon, or by telegram or telefax or overnight courier service, and addressed to the party to whom intended at the following address:

If to Grace: W. R. Grace & Co.-Conn.
Organic Chemicals Division
55 Hayden Avenue
Lexington, Massachusetts 02173
Attention: President

If to Cedar: Mr. William J. Eissler, Jr.
Vice President & General Manager
Organic Chemicals
Cedar Chemical Corporation
24th Floor, Clark Tower
5100 Poplar Avenue
Memphis, Tennessee 38137

19. Default/Termination:

19.1. If either party breaches any of its representations, warranties or undertakings hereunder, becomes insolvent, or commits an act of bankruptcy, or in the event a receiver is appointed for such party, then in such event the other party may terminate this Agreement upon thirty (30) days prior written notice to the party in default; provided, however, if the party in default shall correct the event of default within thirty (30) days after its receipt of such notice, then this Agreement will continue in full force and effect as if no default had occurred.

19.2. Subject to the provisions of Article 9(c), Grace shall have the right to terminate this Agreement at any time upon six (6) months prior written notice to Cedar.

19.3 If Plant Start-Up has not begun within nine (9) months from the Effective Date due to any act or omission of Cedar's in the construction of the Plant (and not due to Grace's failure or inability to supply Raw Materials or inability to arrange for proper off-site disposal of wastes),

Cedar shall pay to Grace a "delay fee" of $25,000 per month payable on the first day of the tenth (10th) month after the Effective Date and the first day of each month thereafter until either initiation of Plant Start-Up or Grace terminates this Agreement as provided in this Article 19. If Plant Start-Up has not begun within fifteen (15) months following the Effective Date as a result of any event or circumstance specified in the last sentence of Article 9(a), then Grace may at its option immediately terminate this Agreement without any obligation to Cedar whatsoever for the payment of any money or the purchase of any Product.

19.4. If due to any reason specified in the last sentence of Article 9(a), Cedar is unable to produce aggregate quantities of Products ordered by Grace for a period of one hundred twenty (120) consecutive days or for a cumulative period of one hundred eighty (180) days in any Contract Year, at least equal to fifty percent (50%) of the demonstrated capacity of the Plant, or if Cedar is unable to produce quantities of Products ordered by Grace for a period of two hundred forty (240) consecutive days in any Contract Year at least equal to the demonstrated capacity of the Plant, Cedar shall be deemed in default, whereupon Grace may at its option, in accordance with this Article 19, terminate this Agreement without any obligation to Cedar whatsoever for the

payment of any money or the purchase of any Product following the effective date of such termination.

19.5. The provisions of Articles 13 and 14 shall survive any termination of this Agreement.

20. Independent Contractor:

The relationship of Cedar to Grace shall be that of an independent contractor and nothing herein contained shall be construed as creating any other relationship. Cedar shall accept in connection with the work called for hereby exclusive liability for the payment of any taxes or contribution for social security, unemployment insurance, or old age payments, or annuities, or retirement benefits which are measured by wages, salaries, or other remuneration paid by Cedar to any and all persons employed by it in connection with the performance of the work and comply with all valid Federal and State administrative regulations respecting the assumption of liability for any of the aforesaid taxes or contributions. Cedar represents that the agreed compensation above stated included all such taxes or contributions and agrees to indemnify and hold Grace and Grace's directors, officers and employees harmless from and against any and all liability for the delay or failure of Cedar and its subcontractors to pay such taxes or contributions.

21. General:

21.1. This Agreement and the Exhibits attached hereto and the Secrecy Agreement referred to in Article 14 constitute the entire agreement between the parties with regard to the matters contained herein and therein, and there are no understandings or agreements expressed or implied not expressly set forth in said documents. No modification of this Agreement or waiver of any of its provisions shall be effective unless in writing and signed by the party to be bound thereby. Neither party's waiver of any breach of any of the provisions of this Agreement shall be deemed to be a waiver of any subsequent breach of the same nature or any breach of a different nature.

21.2. This Agreement shall be binding upon the parties, their successors and permitted assigns. Any attempted assignment of this Agreement or any part thereof without prior written consent of the other party shall be void, provided, however, either party, without such consent, may assign the same in connnection with the transfer or sale of substantially its entire business to which this Agrement pertains or in the event of its merger or consolidation with another company. Any permitted assignee shall assume all obligations of its assignor under this Agreement. No assignment shall relieve any party of responsibility for the performance of any accrued obligation which such party then has hereunder.

IN WITNESS WHEREOF, Cedar and Grace have executed this Agreement as of the date and year first above appearing.

CEDAR CHEMICAL CORPORATION

By: _____

W. R. GRACE & CO.

By: _____

INDEX OF EXHIBITS

Exhibit A	Process Flow Sheets		D-078FS - 01 to 07
	P&ID's		D-078PD - 01 to 11
Exhibit B	Site Plan		
Exhibit C	Target Product Specifications	C-1	TA
		C-2	2-AB
		C-3	AMP
Exhibit D	Process Description	D-1	TN/TA
		D-2	2 NB/2 AB
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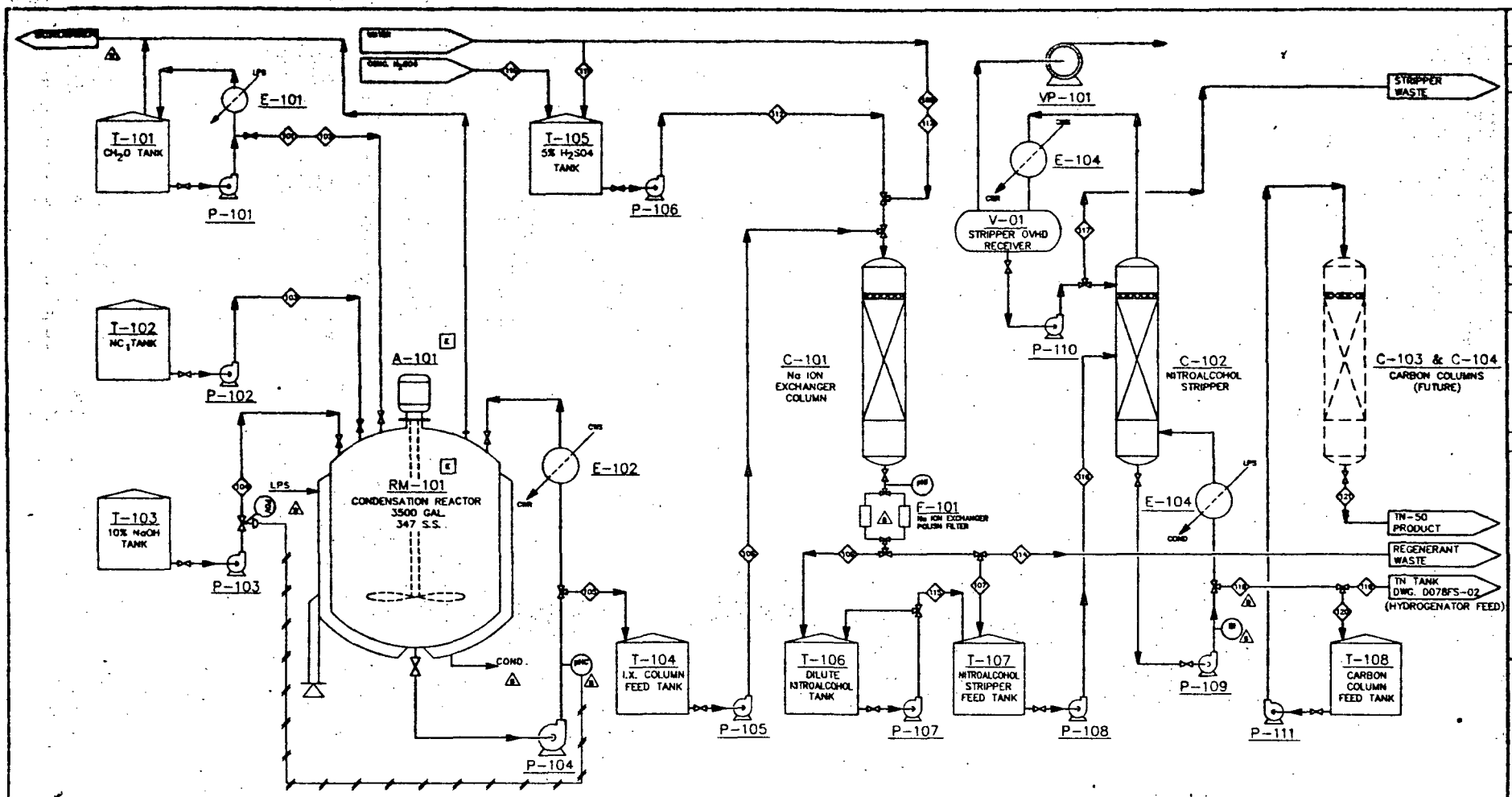
Exhibit E	Raw Material Specification E-1 - Tris (Hydroxy Melthyl) Amino Methane E-2 - Racemic 2-Amino-1-Butanol E-3 - 2-Amino-2-Methyl-1-Propanol		
Exhibit F	Raw Material Assay Procedures		F1 to F6
Exhibit G	Nitroparaffin Derivatives Specification Test Procedurs		NPDST No. 01 to 08

EXHIBIT A

D - 078FS - 01 TO 07

D - 078PD - 01 TO 11



LBS/BATCH — (INITIAL CHARGE) (METERED OVER 3 HOUR PERIOD) — FORWARD WASH — LBS/DAY (1.4 BATCHES/DAY) BACKWASH & RINSE

STREAM / COMPONENT	MOL. WGT	101	102	103	104	105	106	107	108	109	110	111	112	113	114	115	116	117	118	119
CH2O	30.0	684.0	9699.0	0.0	0.0	156.0	229.7	226.5	0.0	0.0	0.0	0.0	0.0	0.0	0.1	3.1	229.7	229.7	0.0	0.0
H2O	18.0	840.0	11904.0	7.0	336.0	13087.0	19320.1	19595.1	3140.5	2892.9	12.6	3197.4	3210.0	14654.2	17861.0	2892.9	22488.0	10954.3	11533.7	11533.7
CH3OH	32.0	31.0	441.0	0.0	0.0	472.0	696.4	686.9	0.0	9.4	0.0	0.0	0.0	0.0	0.1	9.4	696.4	689.0	7.4	7.4
NC1	61.0	0.0	0.0	6781.0	0.0	68.0	100.1	99.0	0.0	1.1	0.0	0.0	0.0	0.0	TRACE	1.1	100.1	59.0	41.1	41.1
NC2	75.0	0.0	0.0	250.0	0.0	3.1	4.6	4.5	0.0	0.1	0.0	0.0	0.0	0.0	TRACE	0.1	4.6	2.6	2.0	2.0
2-NC3	89.0	0.0	0.0	36.0	0.0	0.5	0.6	0.6	0.0	0.0	0.0	0.0	0.0	0.0	TRACE	0.0	0.6	0.4	0.2	0.2
NaOH	40.0	0.0	0.0	0.0	37.0	37.0	54.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
TN	151.1	0.0	0.0	0.0	0.0	16462.0	24302.1	23969.2	0.0	326.6	0.0	0.0	0.0	0.0	18.9	326.6	24384.3	246.5	24137.8	24137.8
NMPD	135.1	0.0	0.0	0.0	0.0	439.0	647.9	638.4	0.0	9.0	0.0	0.0	0.0	0.0	0.5	9.0	646.8	13.2	634.2	634.2
NMP	119.1	0.0	0.0	0.0	0.0	47.0	68.5	67.4	0.0	1.1	0.0	0.0	0.0	0.0	TRACE	1.1	68.5	2.1	66.4	66.4
H2SO4	98.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	168.6	0.0	168.6	0.0	101.1	0.0	0.0	0.0	0.0	0.0
Na2SO4	142.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	98.0	0.0	0.0	0.0	0.0	0.0
OTHER		0.0	0.0	64.0	0.0	341.0	503.6	496.2	0.0	7.1	0.0	0.0	0.0	0.0	0.3	7.1	503.6	0.0	503.6	503.6
TOTAL		1555.0	22044.0	7138.0	373.0	31112.6	45926.4	45784.0	3140.5	3247.1	181.2	3197.4	3378.8	14854.2	18078.1	3250.2	49122.6	12196.8	36926.3	36926.3
VOLUME (gal)		166.5	2360.2	793.7	40.2	3000.3	4429.0	4432.1	376.6	372.4	12.2	383.4	392.9	1757.1	2136.9	372.7	4797.1	1471.3	3400.2	3400.2
TEMP (deg. C)		35	35	20	20	35	30	30	20	20	20	20	20	20	20	20	30	60	60	60
PRESSURE (mm Hg)		760	760	760	760	760	760	760	760	760	760	760	760	760	760	760	760	140	140	140
DENSITY (lb/gal)		9.34	9.34	9.47	9.29	10.37	10.37	10.33	8.34	8.72	14.81	8.34	8.60	8.34	8.46	8.72	10.24	8.29	10.86	10.86

APPROVED
W.R. GRACE - CONN
BY: [signature] DATE: 4/3/89

CEDAR CHEMICAL

TN Process Flowsheet

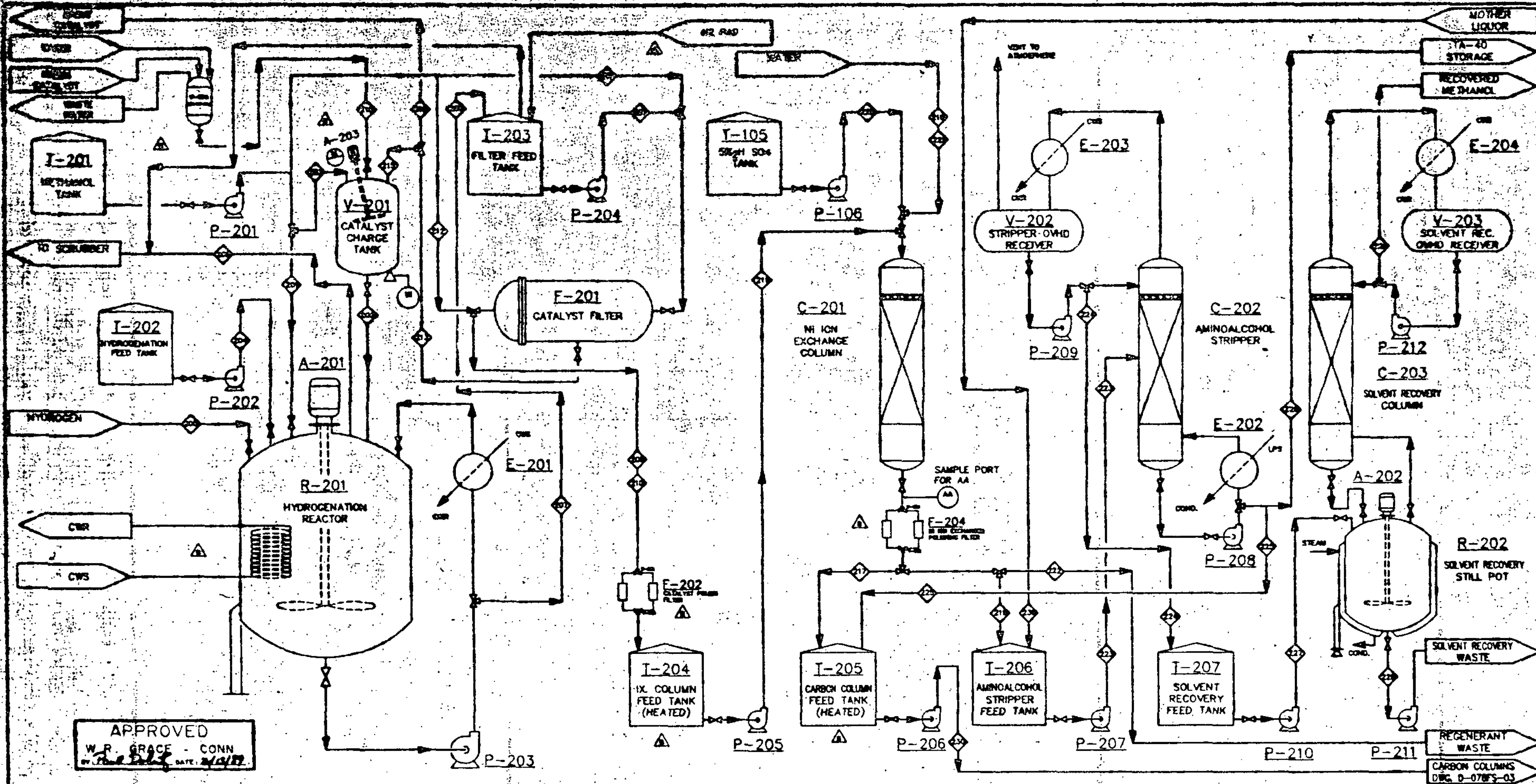
Nitroparaffin Derivatives Project

DELTA PROCESS MANAGEMENT INC.

3/24/88 TTB

NONE

D-078FS-01 B



APPROVED
W.R. GRACE - CONN
BY: [signature] DATE: [illegible]

LBS/BATCH: (INITIAL CHARGE) 201–202; (RINSE) 203; (METERED) 204; (METERED) 205

LBS/DAY (2.88 BATCHES/DAY); (FORWARD WASH); (BACKWASH & RINSE)

	MW	201	202	203	204	205	206	207	207	208	209	210	211	212	213	214
H2O	18.0	34.0	150.0	2.0	3402.0			5494.0	18673.3	17755.7	57.9	961.8	18617.5	131.7	245.5	24.2
CH3OH	32.0	2248.0	2695.0	131.0	2.1			5076.0	17251.1	16404.0	3814.7	3154.2	19550.2	8669.3	10177.9	1017.7
NC1	61.0				12.2											
NC2	75.0				0.6											
2-NC3	89.0				0.1											
TN	181.1				7092.0											
NMPD	135.1				187.0											
NMP	119.1				20.0											
H2SO4	98.0															
Na2SO4	142.2															
TA	121.1		84.0					9423.0	18432.0	17526.0		815.4	18341.4		90.5	9.1
AMPD	125.1		0.8					165.0	561.5	534.1		25.3	559.4		2.7	0.3
AMP	89.1		0.1					14.0	47.4	48.3		2.1	47.4		0.2	TRACE
RANEY NI			850.0					850.0	2889.8						2889.8	288.7
H2	2.0					326.0	9.0									
NH3	17.0						1.0									
NiSO4	154.7															
OTHER			1.1		148.0		13.0	283.0	960.8	913.4		42.1	955.5			0.4
TOTAL		2282.0	3721.0	133.0	10864.0	328.0	23.0	17305.0	58815.8	53178.4	3872.7	4900.9	58079.5	8800.9	13406.8	1340.4
VOLUME (gal)		344.2	635.7	20.1	1000.4			1910.0	6491.8	6077.6	584.1	636.5	6706.6	1327.4	1582.8	158.2
TEMP. (deg C)		25.0	25.0	20.0	25.0			40.0	40.0	40.0	25.0	25.0	40.0	25.0	25.0	25.0
PRESSURE (mm Hg)		760.0	760.0	760.0	760.0			760.0	760.0	760.0	760.0	760.0	760.0	760.0	760.0	760.0
DENSITY (lb/gal)		6.63	8.54	6.63	10.86			9.06	9.06	8.75	6.63	7.70	8.66	6.63	8.47	8.47

	MW	215	216	217	218	219	220	221	222	223	224	225	226	227	228	229	230
H2O	18.0	231.8	288.7	19225.3	7069.0	5878.5	5283.3	32980.2	38841.5	26084.2	23596.3	3572.4	915.5	23596.3	275.0	23321.3	22797.7
CH3OH	32.0	9160.2		18939.5		605.8			12.6	19196.9	19005.1	152.8	39.0	19005.1	18054.9	950.3	19092.6
NC1	61.0																0.0
NC2	75.0																0.0
2-NC3	89.0																0.0
TN	181.1																0.0
NMPD	135.1																0.0
NMP	119.1																0.0
H2SO4	98.0						279.2		230.7								0.0
Na2SO4	142.2																0.0
TA	121.1	81.1		17761.0		567.8			11.6	9068.5	90.6	7143.8	1834.1	90.6		90.6	24904.7
AMPD	125.1	2.4		541.5		16.9			0.3	2462.0	24.2	1940.5	497.3	24.2		24.2	2462.0
AMP	89.1	0.2		46.4		1.5			TRACE	213.9	2.1	188.6	43.2	2.1		2.1	214.9
RANEY NI		2601.1	288.7														0.0
H2	2.0																0.0
NH3	17.0																0.0
NiSO4	154.7								78.9								0.0
OTHER		4.2	0.0	698.5		29.5			23.5	2869.7	28.4	2260.6	580.5	28.4		28.4	3157.3
TOTAL		12081.0	577.3	37410.5	7069.0	7100.0	5562.5	32980.2	39203.2	61895.2	42746.8	15238.9	3909.5	42746.8	18329.8	24417.0	72649.4
VOLUME (gal)		1426.3	49.5	4637.1	847.6	843.2	644.9	3955.4	4650.4	7439.3	5639.4	1520.8	390.2	5639.4	2764.7	2945.4	8126.3
TEMP. (deg C)		25.0	25.0	40.0	20.0	20.0	20.0	20.0	20.0	20.0	100.0	100.0	100.0	100.0	65.0	65.0	60.0
PRESSURE (mm Hg)		760.0	760.0	760.0	760.0	760.0	760.0	760.0	760.0	760.0	760.0	760.0	760.0	760.0	760.0	760.0	760.0
DENSITY (lb/gal)		8.47	11.67	8.65	8.34	8.42	8.60	8.34	8.43	8.32	7.58	10.02	10.02	7.58	6.63	8.29	8.94

CEDAR CHEMICAL CO.
WEST HELENA, ARKANSAS

TA PROCESS FLOWSHEET PAGE 1
300,000 LB/MONTH PRODUCTION RATE

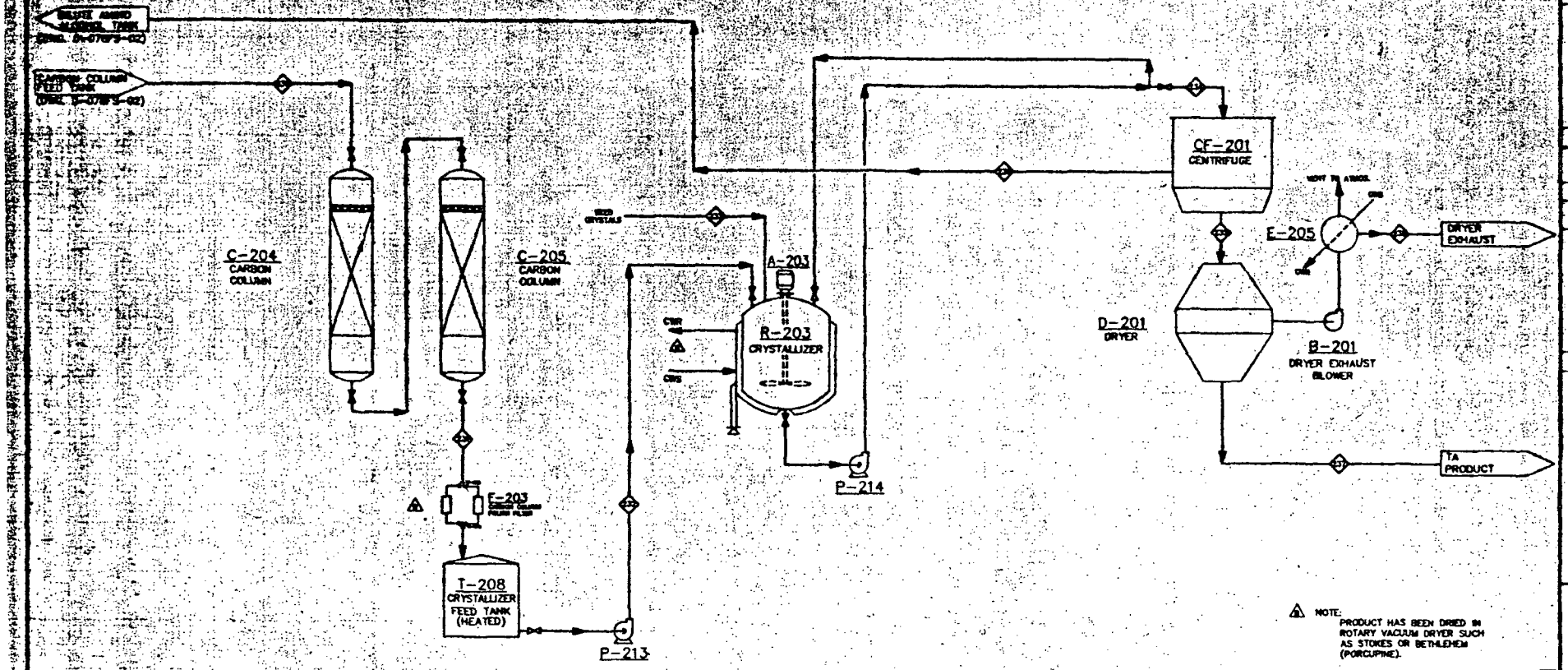
NITROPARAFFIN DERIVATIVES

DELTA PROCESS MANAGEMENT INC.

3-31-88

NONE

D078FS02



⚠ NOTE:
PRODUCT HAS BEEN DRIED IN ROTARY VACUUM DRYER SUCH AS STOKES OR BETHLEHEM (PORCUPINE).

⚠

STREAM NO. / COMPONENT	MOL. WT.	LBS/DAY (3.25 BATCHES/DAY)								
		①	②	③	④	⑤	⑥	⑦	⑧	⑨
H2O	18.0	22797.7	22773.5	22773.5	0.2	22784.0	567.8	28205.7	82.2	485.7
CH3OH	32.0	19092.6	19073.6	19073.6	0.0	19073.6	482.5	18591.1	0.0	482.5
HCl	81.0									
HCl3	75.0									
2-HCl3	88.1									
TN	40.1									
NaPD	138.1									
NaP	119.1									
TA	121.1	24904.7	24880.5	24880.5	49.5	24930.0	16429.3	8500.7	16285.0	164.3
AmPD	125.1	2482.0	2478.9	2478.9	0.1	2478.9	33.7	2445.2	33.4	0.3
AmP	88.1	214.9	214.9	214.9	0.0	214.9	1.7	212.8	2.1	TRACE
H2	2									
OTHER	——	3197.3	3125.7	3125.7	0.8	2178.6	286.6	2840.2	283.4	3.2
TOTAL		72649.4	72547.2	72547.2	50.7	71660.1	17801.6	54795.7	16686.1	1136.0
VOLUME (gal)		8126.3	8114.9	8114.9				6617.8		141.6
TEMPERATURE (deg C)		60	60	60	25	10	10	10	100	25
PRESSURE (mm Hg)		760	760	760	760	760	760	760	760	760
DENSITY (lb/gal)		8.94	8.94	8.94				8.28		8.01

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BY _____ DATE _____

CEDAR CHEMICAL

TA Process Flowsheet, Page 2 (Preliminary)

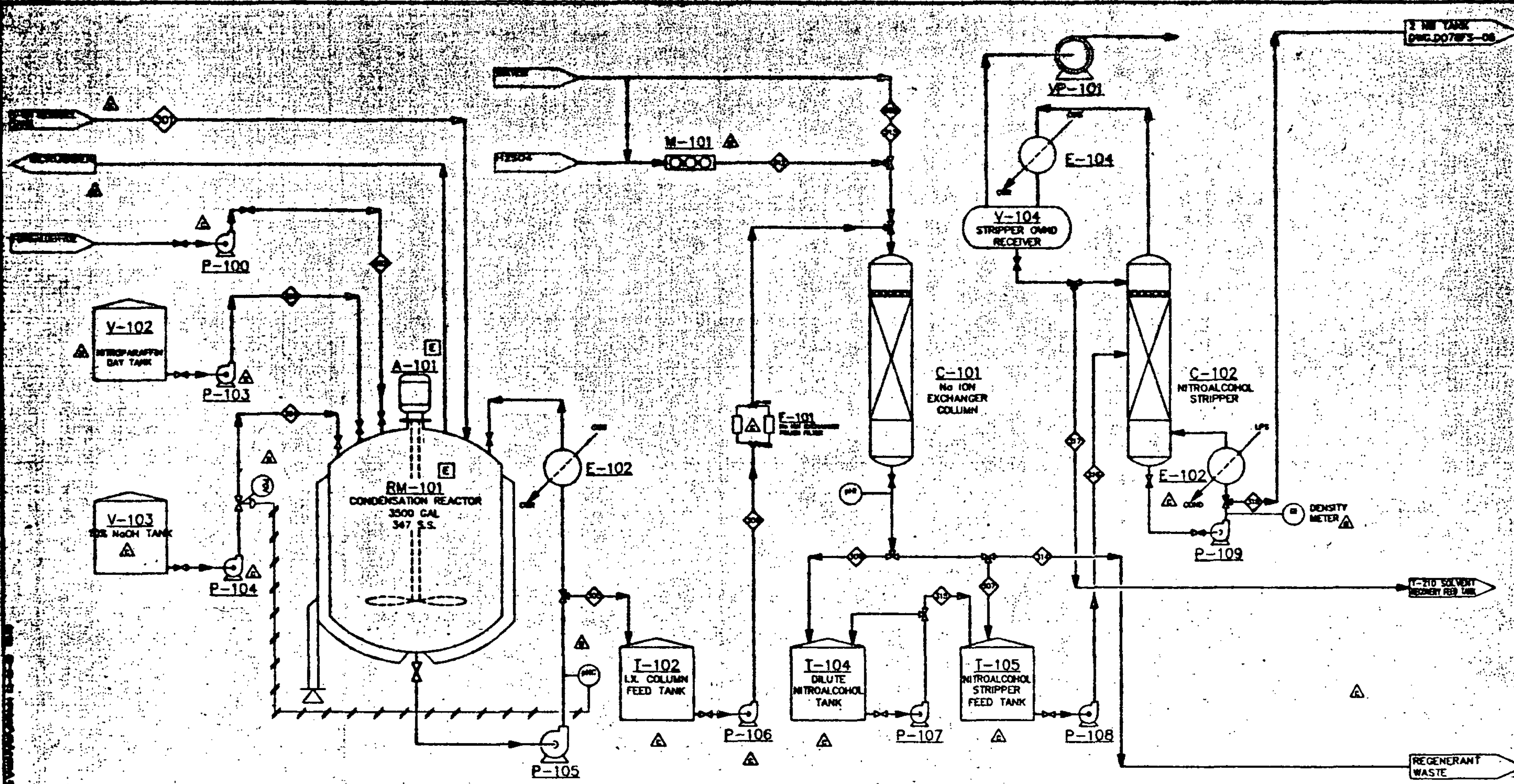
Nitroparaffin Derivatives Project

DELTA PROCESS MANAGEMENT INC.

8/12/88 TTD

MDKE

D-078FS-03 B



COMPONENT / STREAM	MW	301	302	303	304	305	306	307	308	309	310	311	312	313	314	315	316	317	318	319	320	321	322
		LBS/BATCH					LBS/DAY 3.19 BATCHES/DAY																
[illegible]	30.0	65.9	2270.0			86.3	83.7	83.4		0.3					0.0	0.3	83.7	83.7		83.7			
WATER	18.0	6183.4	2700.3	6.7	118.8	2990.7	9664.0	9944.8	1790.3	1536.9	7.3	1822.7	1530.9	5354.0	10179.7	1535.0	11360.3	9961.7	1510.6	9961.7	290.7	941.9	6531.1
METHANOL	32.0		105.2			5304.7	19900.0	19906.1		78.9					5.0	78.5	19978.0	19947.8	30.5	19947.8	19960.2	179.8	606.6
1-NITROPROPANE	89.1			13082.0		6609.1	21097.4	21019.4		77.7					4.3	77.7	21085.1	21082.7	890.4	21082.7		20791.7	210.0
2-NITROPROPANE	89.1			67.4		34.1	109.6	109.2		0.4					0.0	0.4	108.3	107.9	0.7	107.6		108.3	1.3
[illegible]	103.0			67.4		67.4	215.0	214.2		0.8					0.9	0.8	214.9	213.1	1.9	213.1		210.9	2.1
1-NITROBUTANE	103.0			101.8		40.0	154.9	154.3		0.6					0.0	0.6	154.9	154.3	50.6	154.3		153.6	0.8
2-NITROBUTANE	103.0			101.8		61.1	192.9	192.2		0.6					0.0	0.6	192.6	157.8	8.4	157.8		190.7	0.8
[illegible]	[illegible]				12.8	12.8	38.0																0.0
[illegible]	119.1					7990.0	25000.0	24994.6		80.0					4.9	80.0	25063.8	200.8	24833.0	200.8		196.7	50.2
[illegible]	149.1					547.3	1764.9	1728.4		0.2					0.3	0.2	1744.6	0.0	1744.6	0.0		0.0	0.0
[illegible]	119.1					44.6	140.3	141.7		0.5					0.0	0.5	142.2	0.4	141.8	0.4		0.3	0.1
[illegible]	131.0					63.7	203.0	202.3		0.7					0.0	0.7	203.0	0.0	202.0	0.0		0.0	0.0
[illegible]	161.0					3.9	12.5	12.4		0.0					0.0	0.0	12.3	0.0	12.0	0.0		0.0	0.0
[illegible]	133.0					96.7	306.1	306.4		0.7					0.0	0.7	306.1	0.0	306.1	0.0		0.0	0.0
SULFURIC ACID	98.0										98.3		98.3		47.4								0.0
SODIUM SULFATE	142.0														76.6								0.0
[illegible]				60.7		61.7	196.7	196.6		0.7					0.0	0.7	196.7	0.0	196.7	0.0		0.0	0.0
TOTAL		6357.3	5100.2	13486.6	129.1	25029.2	79800.5	79770.3	1790.3	1790.9	103.9	1822.7	1628.3	5354.0	10300.0	1790.9	81563.2	81729.8	79833.7	81729.5	19246.9	22680.6	9710.9
VOLUME, GAL.		949.1	781.0	1626.9	13.9	2997.5	9567.0	9564.3	214.7	214.0	7.0	219.6	224.0	1001.0	1217.1	214.0	9700.0	9770.9	3133.6	9770.9	2916.2	2749.1	1107.1
TEMP., deg. C		20	30	50	20	25	25	30	20	20	20	20	20	20	20	20	30	84	71	84	65	75	75
PRESSURE, mm Hg		760	760	760	760	760	760	760	760	760	760	760	760	760	760	760	760	140	140	760	760	760	760
DENSITY LB/GAL		6.6	6.6	8.29	9.29	8.35	8.38	8.36	8.34	8.34	14.81	8.34	8.6	8.34	8.47	8.34	8.38	7.64	9.62	7.64	6.6	8.26	8.18

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W.R. GRACE - CONN
BY: [signature] DATE 6/13/89

CEDAR CHEMICAL

2NB Process Flowsheet

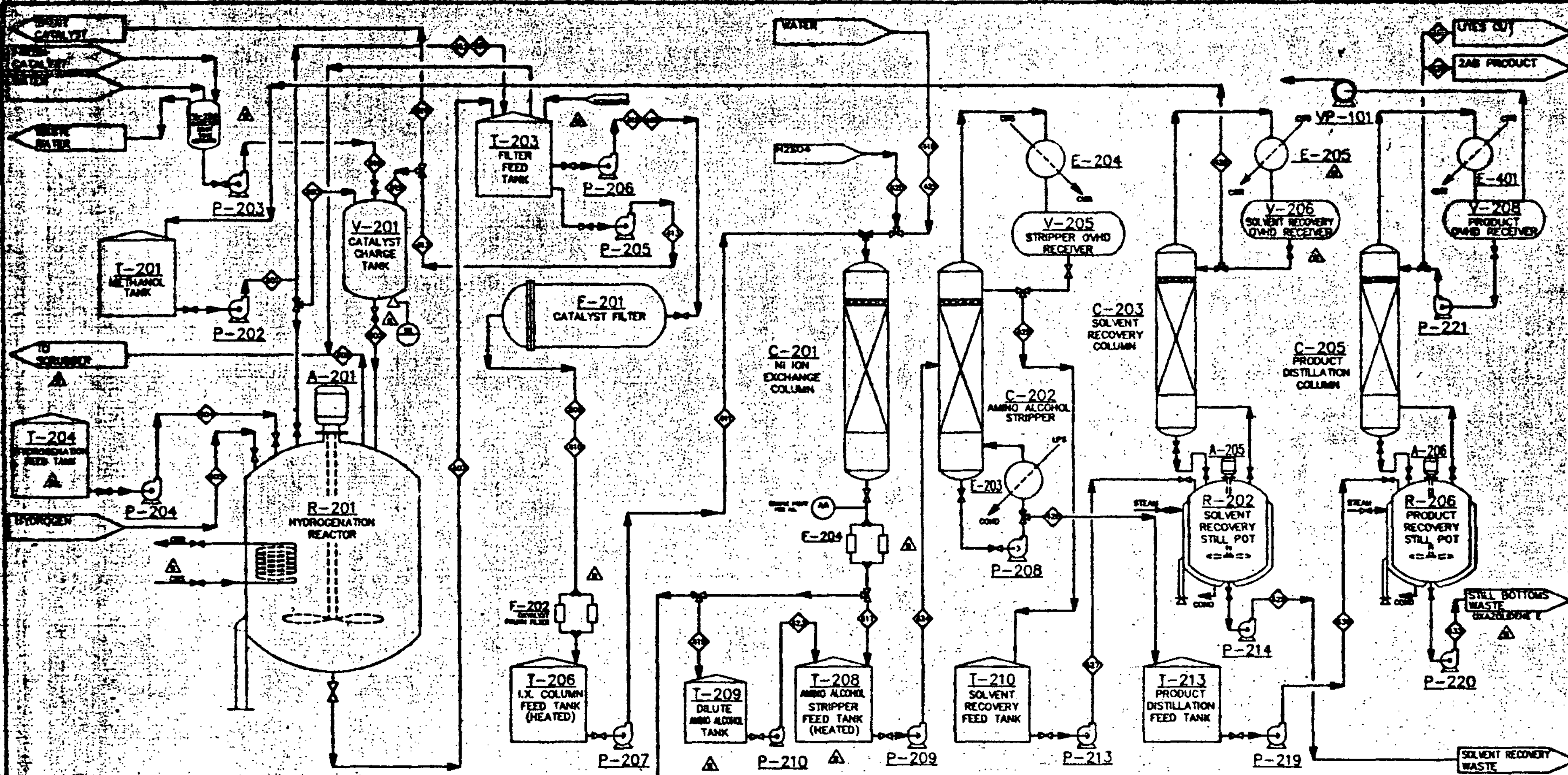
Nitroparaffin Derivatives Project

DELTA PROCESS MANAGEMENT INC.

DATE 6/12/89

SCALE NONE

B-078FS-04 C



* Components with asterisk only found in Stream 404.

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BY: [signature] DATE: [illegible]

LBS/BATCH — LBS/DAY (3.86 BATCHES / DAY)

COMPONENT STREAM	MW	401	402	403	404	405	406	407	407	408	409	410	411
WATER	18.0	0.3	100.0	1.0	476.3			3100.0	9961.0	9961.0	40.7	531.0	10113.4
METHANOL	32.0	413.9	3007.5	96.3	9.9			2407.0	7983.3	7983.1	2073.6	3917.8	9569.3
[illegible]	60.1				270.9								
[illegible]	100.0				9.9								
[illegible]	103.0				10.9								
[illegible]	103.0				1.7								
SULFURIC ACID	98.0												
[illegible]	89/119		23.0		7780.0			5836.5	7040.0	6806.5		700.2	17064.7
[illegible]	119/149		1.0		947.2			411.1	1310.0	1268.1		56.3	1304.4
[illegible]	89/119		0.1		44.5			31.4	100.1	96.3		4.3	99.6
[illegible]	101/131		0.5		63.7			46.2	147.2	140.6		6.3	146.5
[illegible]	131/161		0.0		3.0			3.0	9.6	9.1		0.4	9.5
[illegible]	103/133		0.5		64.5			47.1	150.1	142.9		6.4	148.4
[illegible]			838.3					838.3	3025.5				
[illegible]	2.0					431.6	4.3						
[illegible]	17.0						0.4						
[illegible]	104.7												
OTHER			1.0		61.7		70.7	406.1	1301.0	1230.0		56.0	1294.0
TOTAL		462.1	2700.5	96.3	9387.3	431.6	75.4	2771.1	40718.1	36646.7	2714.3	3437.0	40383.2
VOLUME, GAL.		66	328	10	965			1081	4670	4760	411	478	5171
TEMP., deg. C		85	35	80	25			90	80	40	35	35	25
PRESSURE, mm Hg.		760	760	760	760					760	760	760	760
DENSITY LB/GAL		6.80	6.62	6.60	6.62			6.10	6.16	7.04	6.64	7.24	7.79

COMPONENT STREAM	412	413	414	415	416	417	418	419	420	421	422
WATER	92.0	164.0	10.4	107.0	202.0	10410.4	4475.0	3921.4	2236.0	10013.0	10379.7
METHANOL	6076.0	7111.0	711.0	6460.7		9433.3		136.3			3.2
[illegible]											
[illegible]											
[illegible]											
[illegible]											
SULFURIC ACID									117.7		41.6
[illegible]		86.3	0.4	75.0		17272.0		286.2			5.5
[illegible]		0.2	0.6	5.0		1282.7		21.3			0.4
[illegible]		0.5	0.0	0.4		97.9		1.6			0.0
[illegible]		0.7	0.1	0.6		144.1		2.4			0.0
[illegible]		0.0	0.0	0.0		9.3		0.2			0.0
[illegible]		0.7	0.1	0.6		146.9		2.4			0.1
[illegible]		3025.0	202.5	1822.9	202.5						
[illegible]											
[illegible]											
[illegible]											120.2
OTHER		0.2	0.6	5.0		1884.4		21.1			10.3
TOTAL	6188.9	9390.9	988.8	6488.9	488.1	40087.6	4475.0	4418.9	2384.3	10013.0	10066.4
VOLUME, GAL.	935	1111	111	1009	35	5136	537	534	274	1888	2166
TEMP., deg. C	25	25	35	25	25	25	20	20	20	20	20
PRESSURE, mm Hg.	760	760	760	760	760	760	760	760	760	760	760
DENSITY LB/GAL	6.60	6.46	6.46	6.46	11.67	7.80	8.34	8.35	8.00	8.34	8.30

COMPONENT STREAM	423	424	425	426	427	428	429	430	431	432	433
WATER	3921.4	14337.6	13636.5	799.2	13636.5	150.7	13399.0	799.2	799.2	0.0	0.0
METHANOL	136.3	9660.8	9463.7	96.9	9463.7	9110.2	363.6	96.0	96.9	0.0	0.0
[illegible]											
[illegible]											
[illegible]											
[illegible]											
SULFURIC ACID											
[illegible]	286.2	17060.9	170.6	17363.3	173.6		173.6	17363.3	173.6	16866.1	323.3
[illegible]	21.3	1303.8	15.0	1290.6	13.0		13.0	1290.6	0.0	0.0	1290.5
[illegible]	1.6	99.6	1.0	98.6	1.0		1.0	98.6	40.0	32.5	0.0
[illegible]	2.4	146.5	1.5	146.0	1.5		1.5	146.0	1.4	16.7	129.9
[illegible]	0.2	9.5	0.1	9.4	0.1		0.1	9.4	0.0	0.0	9.4
[illegible]	2.4	149.4	1.5	147.5	1.5		1.5	147.9	0.0	0.0	147.0
[illegible]											
[illegible]											
[illegible]											
[illegible]											
OTHER	21.1	1278.0	22.5	1283.0	22.5		22.0	1283.0	32.2	161.5	1069.2
TOTAL	4412.9	44470.6	23247.4	21233.1	23247.4	9240.0	13890.5	21233.1	1140.6	17117.0	2087.3
VOLUME, GAL.	534	5645.0	3019.1	2638.7	3019.1	1388.0	1698.6	2638.7	141.88	2191.67	323.86
TEMP., deg. C	20	35	75	140	75	65	75	140	150	120	120
PRESSURE, mm Hg.	760	760	760	760	760	760	760	760	[illegible]	200	200
DENSITY LB/GAL	8.26	7.85	7.7	8.04	7.7	6.61	8.28	8.04	[illegible]	7.61	9.14

CEDAR CHEMICAL

2AB PROCESS FLOW SHEET
500,000 LBS/MO. PRODUCTION RATE

Nitroparaffin Derivatives Project

DELTA PROCESS MANAGEMENT INC.

6/17/88

SCALE: NONE

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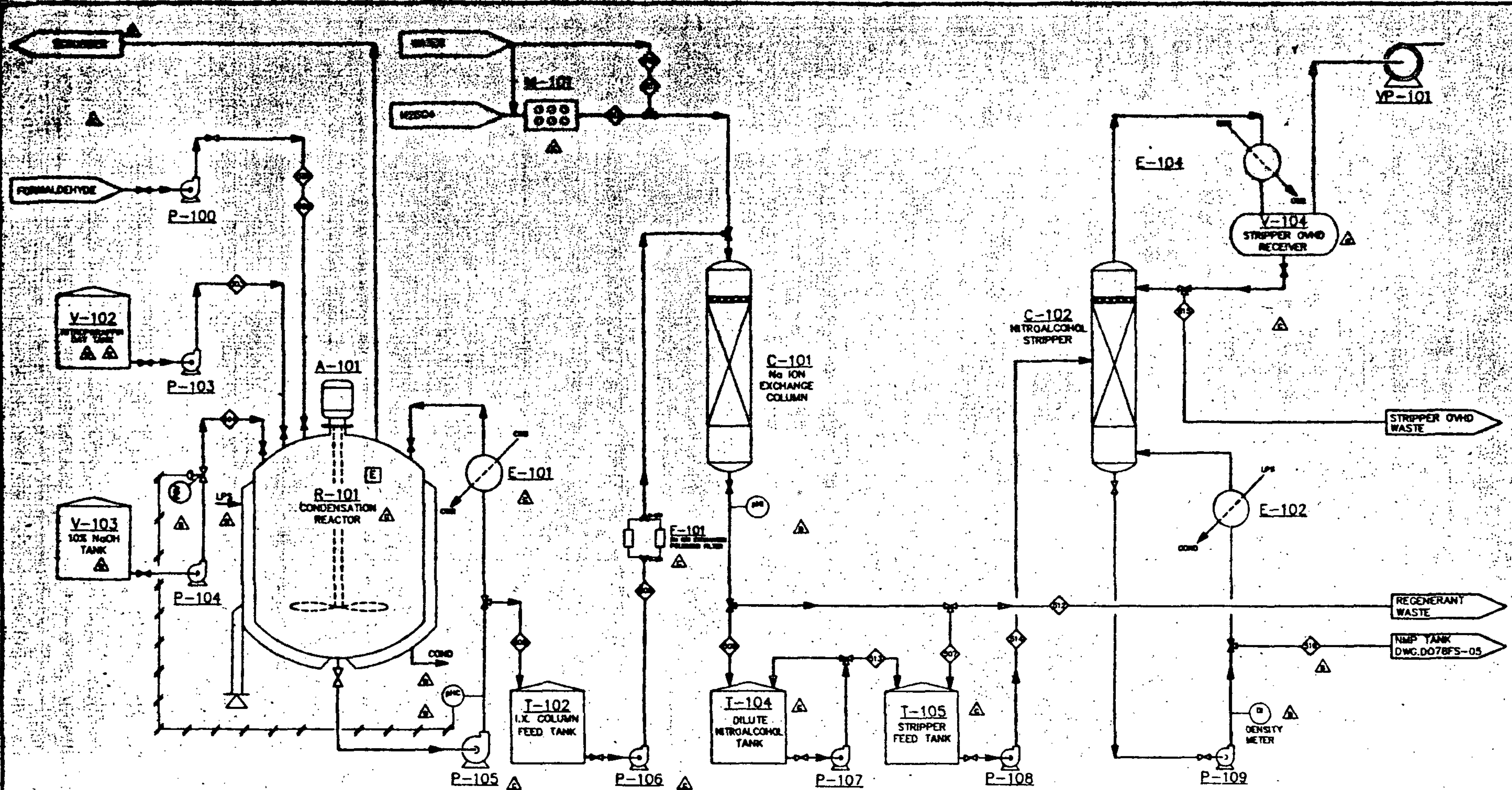


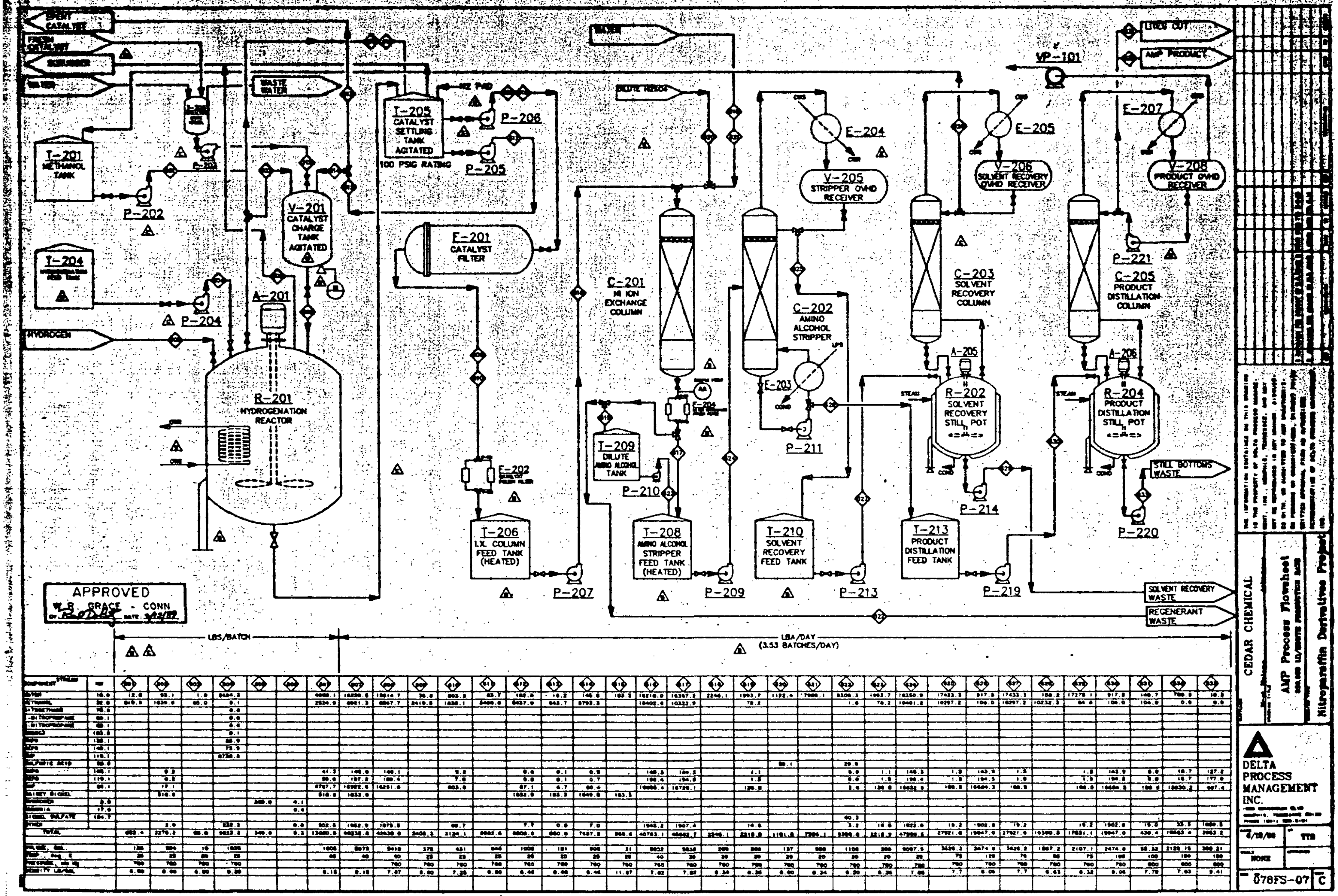
COMPONENT / STREAM	MW	LBS/BATCH 001	002	003	004	005	LBS/DAY (1.33 BATCHES / DAY) 006	007	008	009	010	011	012	013	014	015	016
FORMALDEHYDE	30.0	908.3	4041.2			140.6	185.0	177.7		2.1			0.1	2.1	175.9	175.9	0.0
WATER	18.0	748.6	9941.5	7.4	487.7	7193.5	9207.7	9684.0	2309.0	2116.0	[illegible]	11180.8	13822.8	2116.0	11042.6	2073.4	[illegible]
METHANOL	32.0	87.7	220.1			247.7	317.1	313.1		3.7			0.2	5.7	316.9	316.8	0.3
NITROETHANE	75.0			66.3		1.1	1.4	1.0		0.0			0.0	0.0	1.4	1.4	0.0
1-NITROPROPANE	89.1			125.1		1.6	2.0	2.0		0.0			0.0	0.0	2.0	2.0	0.0
2-NITROPROPANE	89.1			14467.8		182.3	230.0	220.0		2.7			0.3	2.7	230.7	220.4	0.3
[illegible]	105.0			36.8		36.2	47.1	46.5		0.9			0.9	0.9	47.1	46.4	4.8
SODIUM HYDROXIDE	40.0				90.3	90.3	70.0										
NMPD	135.1					186.1	180.6	190.1		2.3			0.1	2.3	196.4	1.8	197.4
NEPD	149.1					204.1	281.3	280.1		3.1			0.2	3.1	281.1	1.3	280.9
NMP	119.1					18802.8	24067.9	23767.1		280.3			12.0	280.3	24091.3	280.5	23810.9
SULFURIC ACID	98.0										128.9		43.4				
SODIUM SULFATE	142.2												188.8				
OTHER				36.8		641.8	821.2	811.0		9.7			0.9	9.7	820.7	0.0	820.7
TOTAL		1383.0	11882.7	14721.4	583.0	27600.2	35409.1	35320.0	2309.0	2427.2	2377.8	11180.8	13807.3	2427.2	37708.0	4081.1	33480.9
VOLUME, GAL.		148.1	1178.0	1790.7	80.6	3080.0	3940.0	3943.6	257.3	288.9	299.7	1344.6	1620.3	288.9	4130.4	480.2	3387.8
TEMP., deg. C		20	30	20	20	80	80	40	20	20	20	20	20	80	35	25	35
PRESSURE, mm Hg.		760	760	760	760	760	760	760	760	712	760	760	760	760	760	760	760
DENSITY LB/GAL		9.34	9.34	8.10	9.29	9.22	9.22	9.10	8.34	8.40	8.6	8.34	8.40	8.40	9.14	8.73	10.24

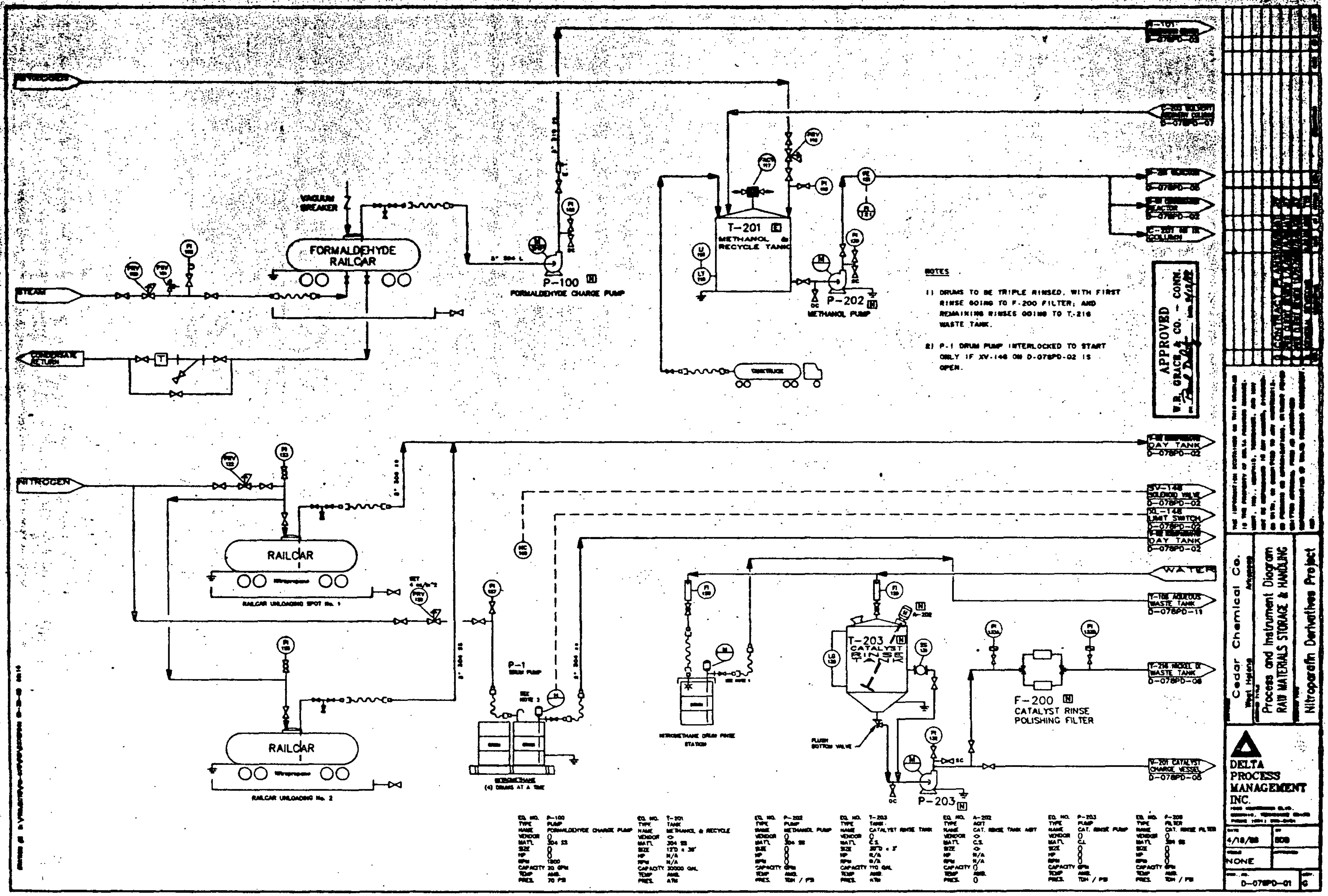
APPROVED
W.R. GRACE - CONN
BY [signature] DATE: 3/15/89

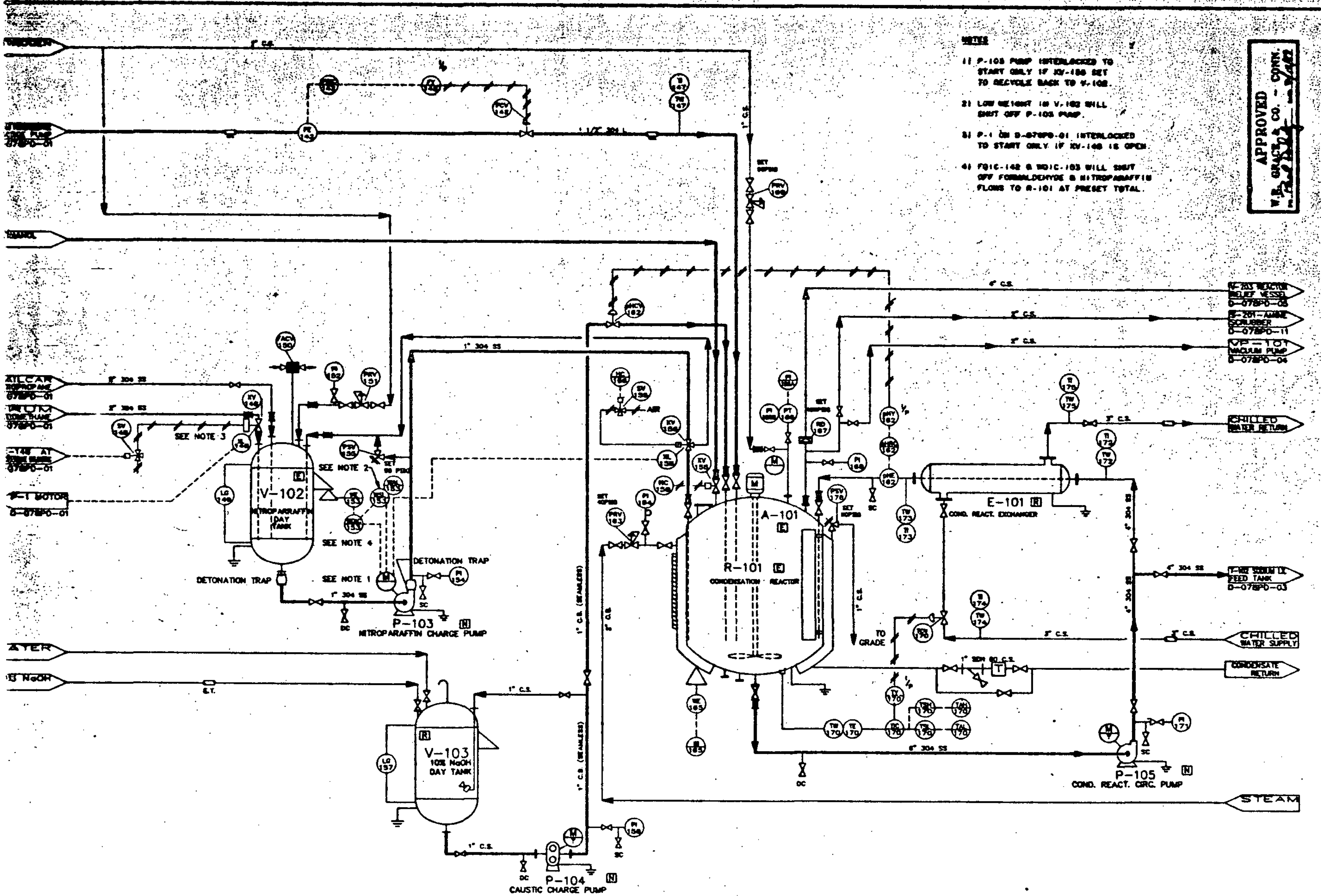
CEDAR CHEMICAL
WEST HELENA ARKANSAS
NMP PROCESS FLOWSHEET
500,000 LB/MONTH PRODUCTION RATE
NITROPARAFFIN DERIVATIVES PROJECT

DELTA PROCESS MANAGEMENT INC.

6/17/88 TTB
NONE
D-078FS-06 C


SPENT CATALYST
FRESH CATALYST
SCRUBBER
WATER
WASTE WATER
HYDROGEN
T-201 METHANOL TANK
P-202
T-204
P-204
T-205 CATALYST SETTLING TANK AGITATED
100 PSIG RATING
N2 PAD
P-206
P-205
V-201 CATALYST CHARGE TANK AGITATED
A-201
R-201 HYDROGENATION REACTOR
F-201 CATALYST FILTER
F-202
T-206 I.X. COLUMN FEED TANK (HEATED)
P-207
DILUTE H2SO4
C-201 Ni ION EXCHANGE COLUMN
T-209 DILUTE AMINO ALCOHOL TANK
P-210
T-208 AMINO ALCOHOL STRIPPER FEED TANK (HEATED)
P-209
E-204
V-205 STRIPPER OVHD RECEIVER
C-202 AMINO ALCOHOL STRIPPER
E-203
P-211
T-210 SOLVENT RECOVERY FEED TANK
P-213
VP-101
E-205
V-206 SOLVENT RECOVERY OVHD RECEIVER
C-203 SOLVENT RECOVERY COLUMN
A-205
R-202 SOLVENT RECOVERY STILL POT
STEAM
P-214
T-213 PRODUCT DISTILLATION FEED TANK
P-219
E-207
V-208 PRODUCT OVHD RECEIVER
P-221
C-205 PRODUCT DISTILLATION COLUMN
A-206
R-204 PRODUCT DISTILLATION STILL POT
P-220
LITES CUT
AMP PRODUCT
STILL BOTTOMS WASTE
SOLVENT RECOVERY WASTE
REGENERANT WASTE
APPROVED
W.R. GRACE - CONN
LBS/BATCH
LBS/DAY (3.53 BATCHES/DAY)
CEDAR CHEMICAL
AMP Process Flowsheet
Nitroparaffin Derivatives Project
DELTA PROCESS MANAGEMENT INC.
078FS-07
C


FORMALDEHYDE RAILCAR
VACUUM BREAKER
STEAM
CONDENSATE RETURN
P-100
FORMALDEHYDE CHARGE PUMP
T-201
METHANOL & RECYCLE TANK
P-202
METHANOL PUMP
NOTES
1) DRUMS TO BE TRIPLE RINSED, WITH FIRST RINSE GOING TO F-200 FILTER; AND REMAINING RINSES GOING TO T-216 WASTE TANK.
2) P-1 DRUM PUMP INTERLOCKED TO START ONLY IF XV-146 ON D-078PD-02 IS OPEN.
APPROVED
W.R. GRACE & CO. - CONN.
NITROGEN
RAILCAR
RAILCAR UNLOADING SPOT No. 1
RAILCAR UNLOADING No. 2
P-1
DRUM PUMP
NITROMETHANE DRUM RINSE STATION
WATER
T-203
CATALYST RINSE TANK
FLUSH BOTTOM VALVE
P-203
F-200
CATALYST RINSE POLISHING FILTER
DAY TANK
D-078PD-02
T-108 AQUEOUS WASTE TANK
D-078PD-11
WASTE TANK
D-078PD-08
Cedar Chemical Co.
Process and Instrument Diagram
RAW MATERIALS STORAGE & HANDLING
Nitroparaffin Derivatives Project
DELTA PROCESS MANAGEMENT INC.
4/18/88
NONE
D-078PD-01



EQ. NO.	V-102	EQ. NO.	P-103	EQ. NO.	V-103	EQ. NO.	P-104	EQ. NO.	R-101	EQ. NO.	A-101	EQ. NO.	E-101	EQ. NO.	P-105
TYPE	VESL	TYPE	PUMP	TYPE	VESL	TYPE	PUMP	TYPE	REACTOR	TYPE	AGIT	TYPE	EXCH	TYPE	PUMP
NAME	NITROP. DAY TANK	NAME	NITRO. CHG PUMP	NAME	10% NaOH DAY TANK	NAME	CAUSTIC CHARGE PUMP	NAME	COND. REACTOR	NAME	COND REACTOR AGIT	NAME	COND REACT. EXCH.	NAME	COND. REACT. CIRC. PUMP
VENDOR	◇	VENDOR	0	VENDOR	◇	VENDOR	0	VENDOR	[illegible]	VENDOR	[illegible]	VENDOR	BEAUMONT	VENDOR	0
MAT'L	316 SS	MAT'L	316 SS	MAT'L	C.S.	MAT'L	C.I.	MAT'L	347 SS	MAT'L	304 SS	MATERIAL	316 SS	MAT'L	316 SS
SIZE	7'-0"D x 8'-0"	SIZE	0	SIZE	2'-0"D x 3'-0"	SIZE	0	SIZE	8'6"D x 8'	SIZE	◇	SIZE	300 S.F.	SIZE	0
HP	N/A	HP	0	HP	N/A	HP	0	HP	N/A	HP	25	HP	N/A	HP	0
RPM	N/A	RPM	0	RPM	N/A	RPM	0	RPM	N/A	RPM	0	RPM	N/A	RPM	0
CAPACITY	2200 GAL	CAPACITY	5 GPM	CAPACITY	100 GAL	CAPACITY	15 GPM	CAPACITY	3600 GAL	CAPACITY	N/A	MM BTUH	1.8 MM BTUH	CAPACITY	300 GPM
TEMP	AMB.	TEMP	AMB.	TEMP	AMB.	TEMP	AMB.	TEMP	AMB.	TEMP	38 C	TEMP	100 C	TEMP	AMB.
PRES.	0	PRES.	160' TDH	PRES.	0	PRES.	70 PSIG	PRES.	180 PSIG INT./FULL VACUUM	PRES.	30 PSIG	PRES.	100 PSIG	PRES.	60' TDH

G CONTRACT PLANS
F PER CLIENT REVIEW
E PER CLIENT REVIEW
D GENERAL REVISIONS

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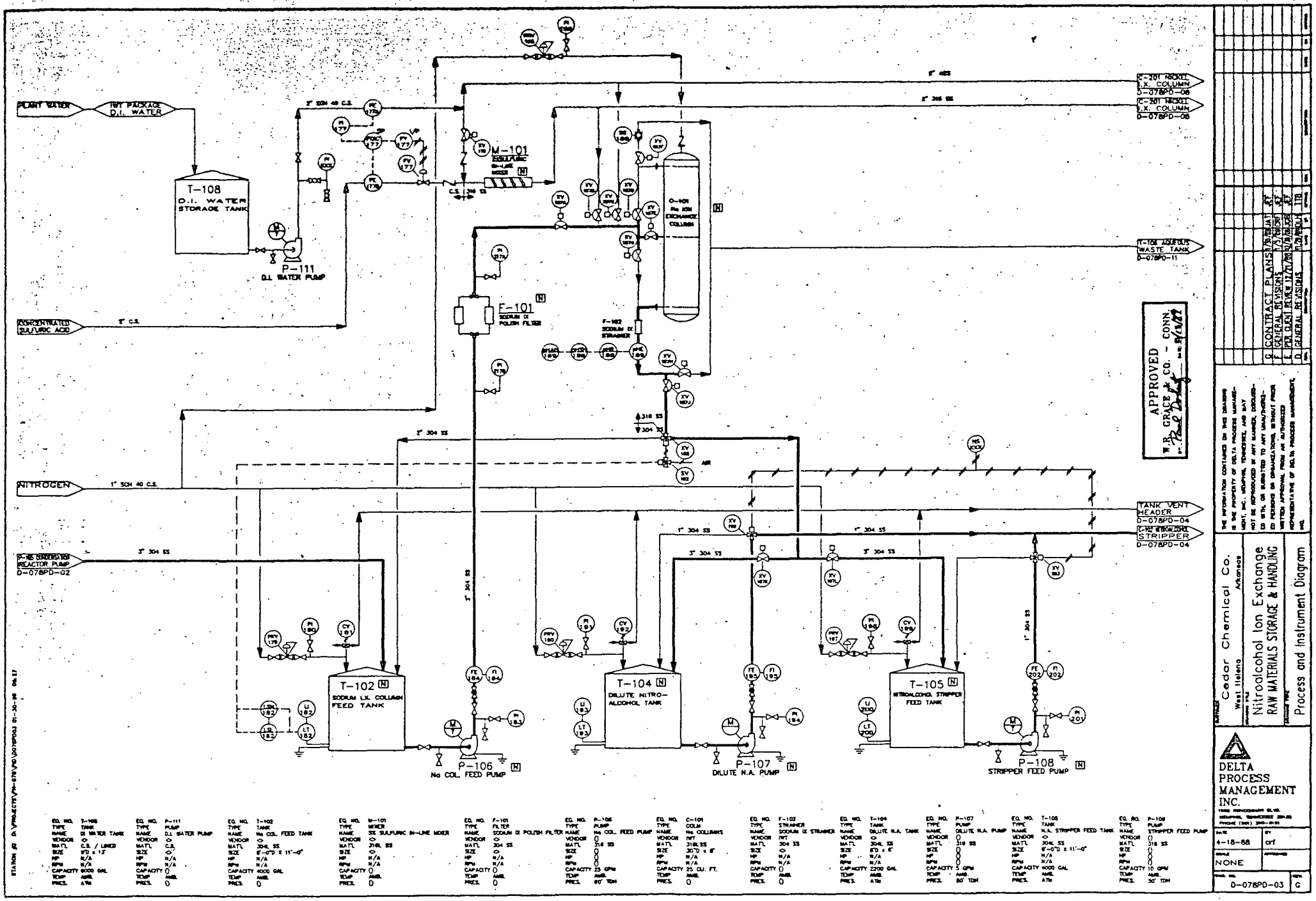
Cedar Chemical Co.
West Helena Arkansas

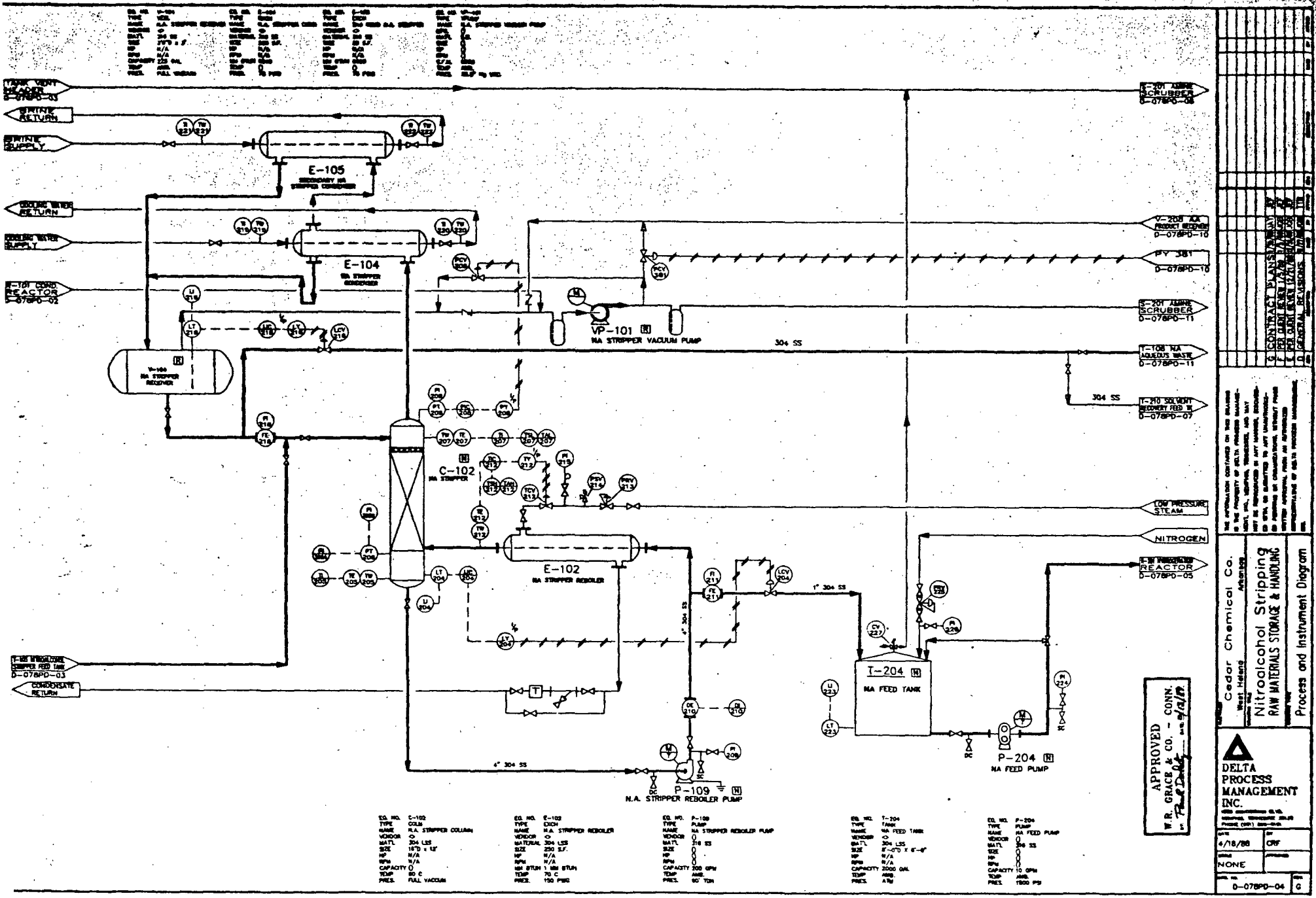
NITROPARAFFIN DERIVATIVES PROJECT
CONDENSATION REACTION

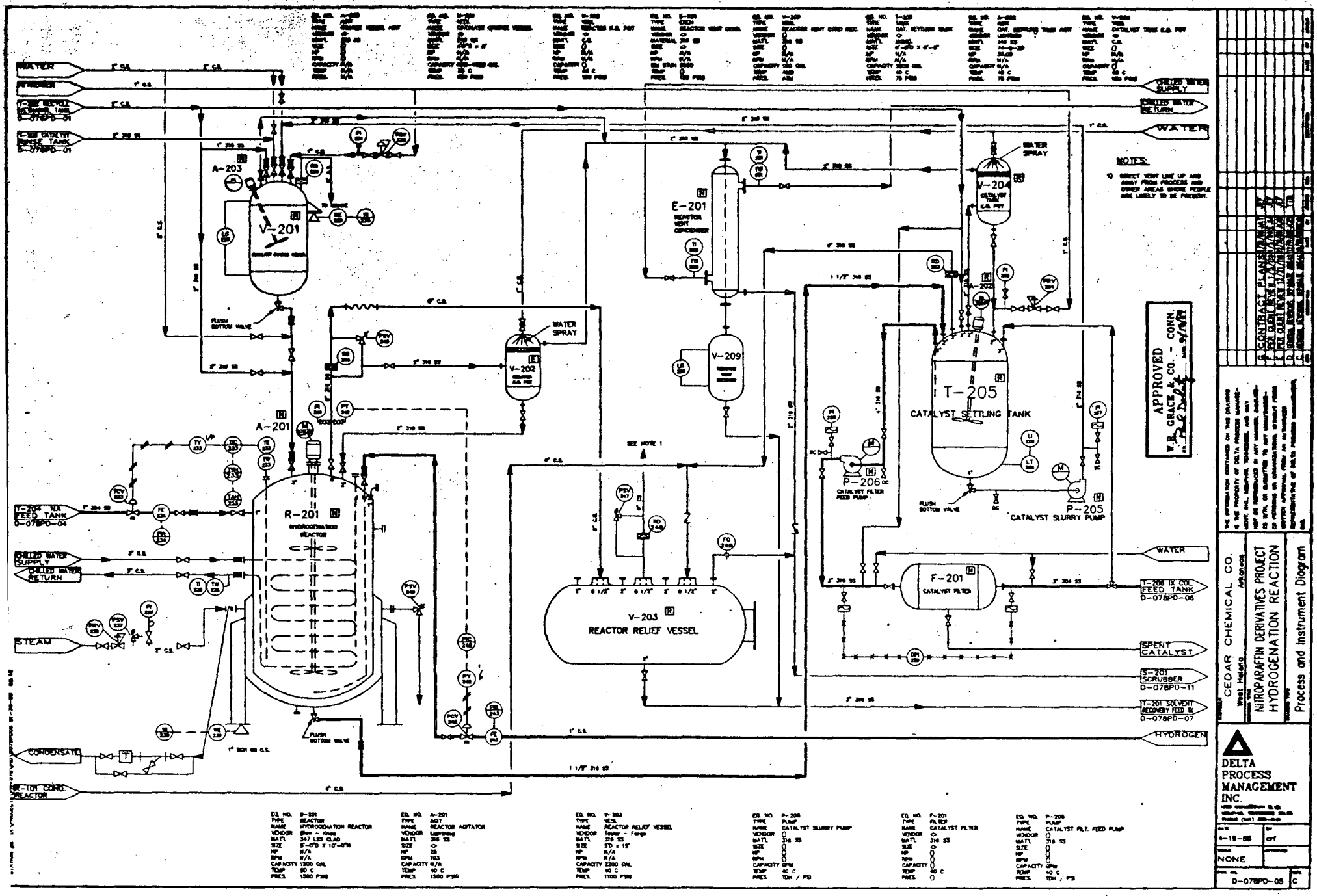
PROCESS AND INSTRUMENT DIAGRAM

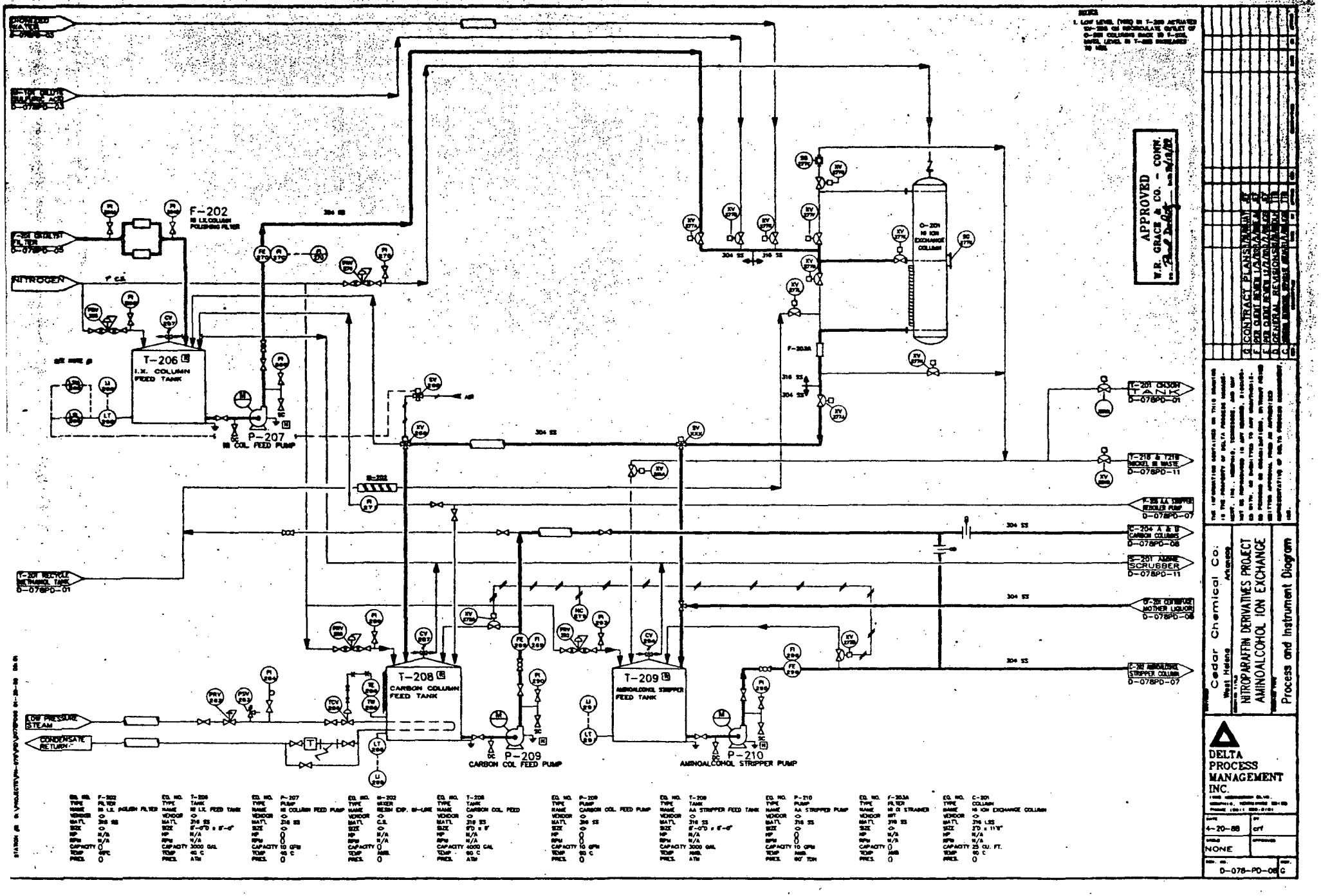
DELTA PROCESS MANAGEMENT INC.

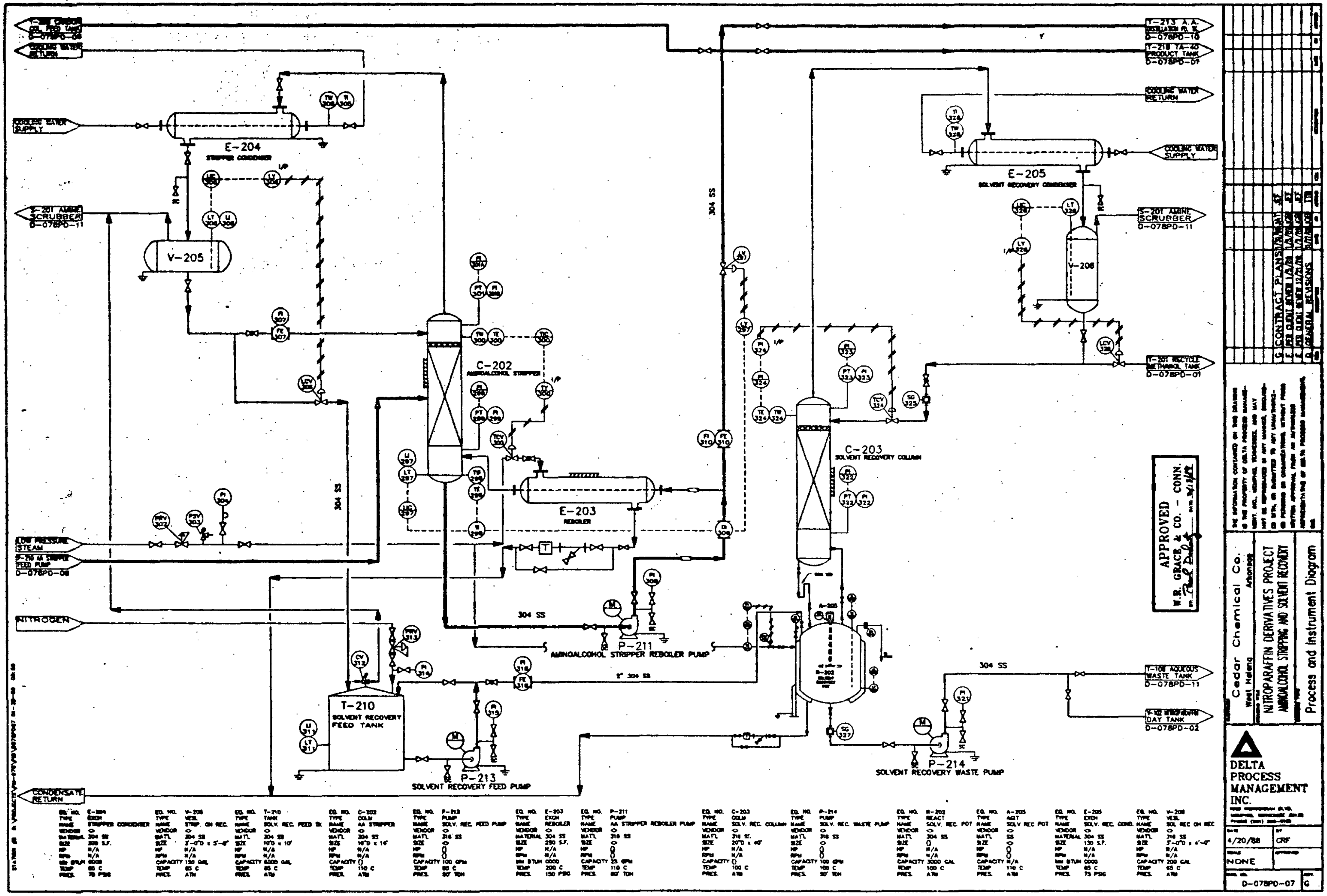
DATE: 4/18/88/ BY: crf
SCALE: NONE
DWG. NO. D-078PD-02 REV. G


PLANT WATER
INT PACKAGE D.I. WATER
T-108
D.I. WATER STORAGE TANK
P-111
D.I. WATER PUMP
CONCENTRATED SULFURIC ACID
M-101
SULFURIC IN-LINE MIXER
F-101
SODIUM IX POLISH FILTER
P-101
SODIUM IX CHROMATOGRAPHIC COLUMN
F-102
SODIUM IX STRAINER
NITROGEN
P-105 SODIUM IX REACTOR PUMP D-078PD-02
C-201 NICKEL I.X. COLUMN D-078PD-06
C-201 NICKEL I.X. COLUMN D-078PD-06
T-108 AQUEOUS WASTE TANK D-078PD-11
TANK VENT HEADER D-078PD-04
C-102 NITROALCOHOL STRIPPER D-078PD-04
T-102
SODIUM I.X. COLUMN FEED TANK
T-104
DILUTE NITRO-ALCOHOL TANK
T-105
NITROALCOHOL STRIPPER FEED TANK
P-106
Na COL. FEED PUMP
P-107
DILUTE N.A. PUMP
P-108
STRIPPER FEED PUMP
APPROVED
W.R. GRACE & CO. - CONN.
Cedar Chemical Co.
West Helena
Arkansas
Nitroalcohol Ion Exchange
RAW MATERIALS STORAGE & HANDLING
Process and Instrument Diagram
DELTA PROCESS MANAGEMENT INC.
4-18-88
NONE
D-078PD-03

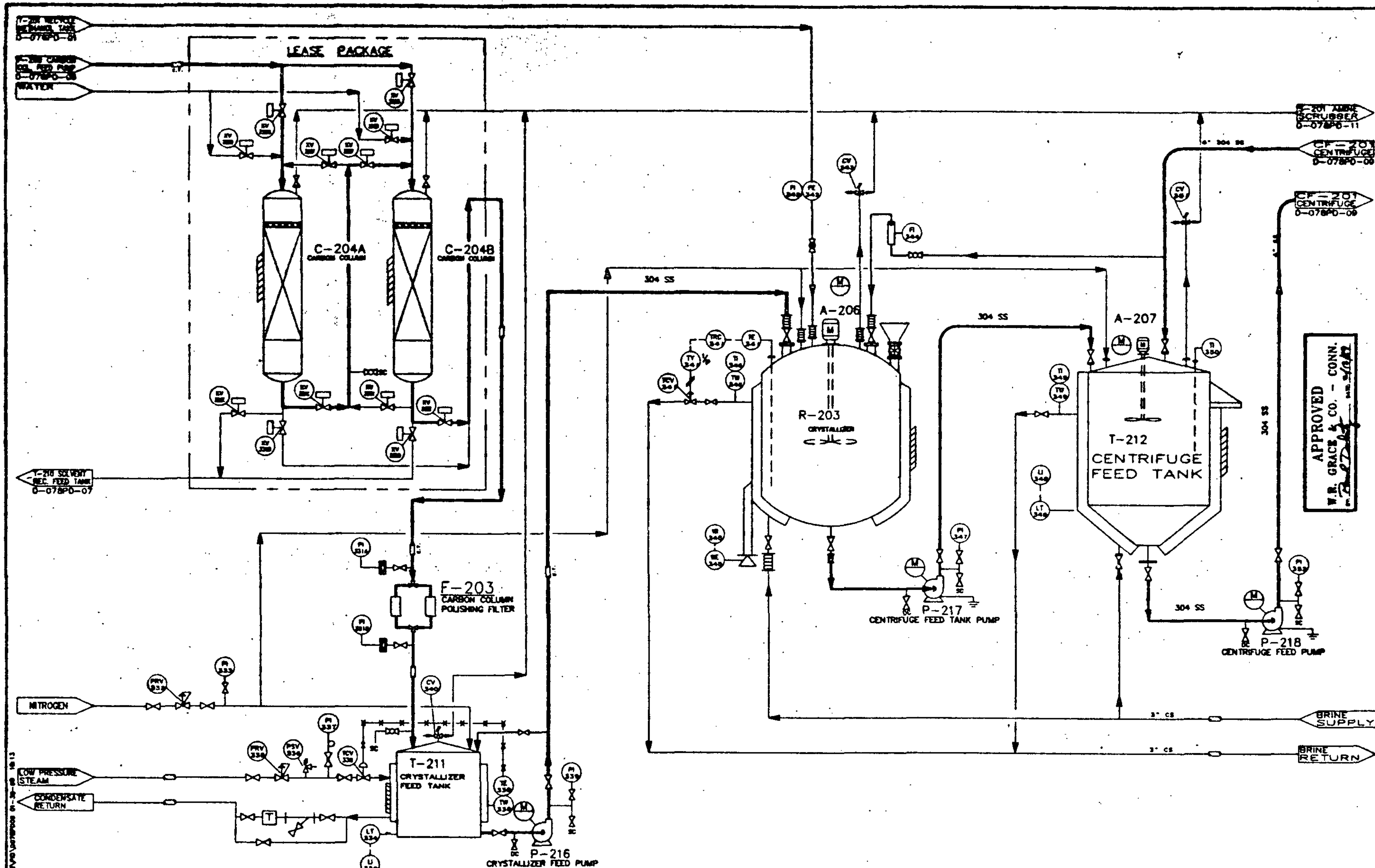

E-105
E-104
VP-101
304 SS
C-102
E-102
I-204
P-204
P-109
1" 304 SS
4" 304 SS
4" 304 SS
LOW PRESSURE STEAM
NITROGEN
APPROVED
W.R. GRACE & CO. - CONN.
Cedar Chemical Co.
Nitroalcohol Stripping
RAW MATERIALS STORAGE & HANDLING
Process and Instrument Diagram
DELTA PROCESS MANAGEMENT INC.
4/18/88
NONE
D-078PD-04


WATER
CHILLED WATER SUPPLY
CHILLED WATER RETURN
WATER
A-203
V-201
E-201
REACTOR VENT CONDENSER
V-20
WATER SPRAY
NOTES:
A-201
V-202
WATER SPRAY
V-209
T-205
CATALYST SETTLING TANK
APPROVED
W.R. GRACE & CO. - CONN.
R-201
HYDROGENATION REACTOR
P-206
CATALYST FILTER FEED PUMP
P-205
CATALYST SLURRY PUMP
F-201
CATALYST FILTER
V-203
REACTOR RELIEF VESSEL
SEE NOTE 1
T-204 NA FEED TANK D-078PD-04
CHILLED WATER SUPPLY
CHILLED WATER RETURN
STEAM
CONDENSATE
WATER
T-206 IX COL FEED TANK D-078PD-06
SPENT CATALYST
R-201 SCRUBBER D-078PD-11
T-201 SOLVENT RECOVERY FEED TK D-078PD-07
HYDROGEN
FLUSH BOTTOM VALVE
CEDAR CHEMICAL CO.
West Helena
Arkansas
NITROPARAFFIN DERIVATIVES PROJECT
HYDROGENATION REACTION
Process and Instrument Diagram
DELTA PROCESS MANAGEMENT INC.
4-19-88
NONE
D-078PD-05
C


F-202
T-206
I.X. COLUMN FEED TANK
P-207
T-208
CARBON COLUMN FEED TANK
P-209
CARBON COL FEED PUMP
T-209
AMINOALCOHOL STRIPPER FEED TANK
P-210
AMINOALCOHOL STRIPPER PUMP
NITROGEN
LOW PRESSURE STEAM
CONDENSATE RETURN
APPROVED
W.R. GRACE & CO. - CONN.
Cedar Chemical Co.
West Helena
Arkansas
NITROPARAFFIN DERIVATIVES PROJECT
AMINOALCOHOL ION EXCHANGE
Process and Instrument Diagram
DELTA PROCESS MANAGEMENT INC.
D-078-PD-06
G



E-204
STRIPPER CONDENSER
V-205
C-202
AMINOALCOHOL STRIPPER
E-203
REBOILER
P-211
AMINOALCOHOL STRIPPER REBOILER PUMP
T-210
SOLVENT RECOVERY FEED TANK
P-213
SOLVENT RECOVERY FEED PUMP
C-203
SOLVENT RECOVERY COLUMN
E-205
SOLVENT RECOVERY CONDENSER
V-206
P-214
SOLVENT RECOVERY WASTE PUMP
COOLING WATER SUPPLY
COOLING WATER RETURN
S-201 AMINE SCRUBBER
D-078PD-11
LOW PRESSURE STEAM
NITROGEN
CONDENSATE RETURN
T-201 RECYCLE METHANOL TANK
D-078PD-01
T-108 AQUEOUS WASTE TANK
D-078PD-11
T-213 A.A. DISTILLATION FD. TK.
D-078PD-10
T-218 TA-40 PRODUCT TANK
D-078PD-07
D-078PD-08
D-078PD-02
304 SS
2" 304 SS
APPROVED
W.R. GRACE & CO. - CONN.
Cedar Chemical Co.
West Helena Arkansas
NITROPARAFFIN DERIVATIVES PROJECT
AMINOALCOHOL STRIPPING AND SOLVENT RECOVERY
Process and Instrument Diagram
DELTA PROCESS MANAGEMENT INC.
4/20/88
CRF
NONE
D-078PD-07
G
CONTRACT PLANS
GENERAL REVISIONS



EQ. NO.	C-204A	C-204B	F-203	T-211	P-216	R-203
TYPE	COLM	COLM	FILTER	TANK	PUMP	REACTOR
NAME	CARBON COLUMN	CARBON COLUMN	CARBON POLISH FILTER	CRYSTALLIZER FEED TANK	CRYSTALLIZER FEED PUMP	CRYSTALLIZER
VENDOR	◇	◇	◇	◇	◇	◇
MAT'L	C.S./LINED	C.S./LINED	304 SS	304 SS	316 SS	GLS
SIZE	5'-0"D x 6'-6"	5'-0"D x 6'-6"	◇	6'-0"D x 11'-6"	◇	8'D x8'
HP	N/A	N/A	N/A	N/A	()	N/A
RPM	N/A	N/A	N/A	N/A	()	N/A
CAPACITY	()	()	()	4000 GAL	200 GPM	3000 GAL
TEMP	80 C	80 C	80 C	80 C	80 C	80 C
PRES.	130 PSIG	130 PSIG	()	ATM	50' TDH	ATM

EQ. NO.	A-206	P-217	T-212	P-218	A-207
TYPE	AGIT	PUMP	TANK	PUMP	AGIT
NAME	CRYSTALLIZER	CENTRIFUGE FEED PUMP	CENTRIFUGE FEED TANK	CENTRIFUGE FEED PUMP	CENTR. TANK
VENDOR	◇	◇	◇	◇	◇
MAT'L	316 SS	316 SS	GLS	316 SS	316 SS
SIZE	◇	◇	8'D x 11'	◇	◇
HP	()	()	N/A	()	()
RPM	()	()	N/A	()	()
CAPACITY	N/A	200 GPM	4000 GAL	150 GPM	N/A
TEMP	80 C	10 C	10 C	10 C	10 C
PRES.	ATM	50' TDH	ATM	50' TDH	ATM

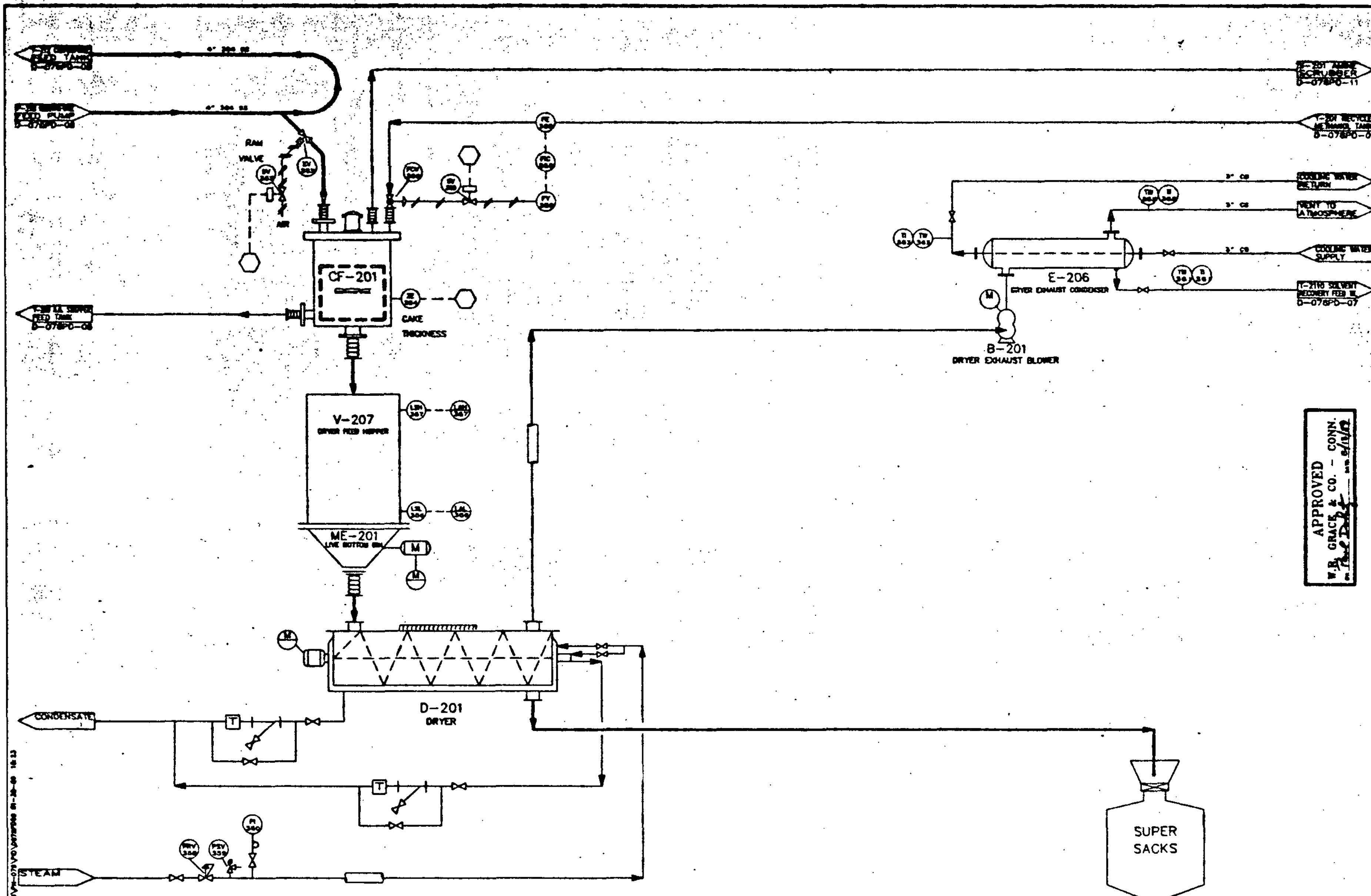
APPROVED W.R. GRACE & CO. - CONN.

G CONTRACT PLANS
F PER CLIENT REVIEW
E PER CLIENT REVIEW
D GENERAL REVISIONS

Cedar Chemical Co.
West Helena Arkansas
NITROPARAFFIN DERIVATIVES PROJECT
CARBON TREATMENT AND CRYSTALLIZATION
Process and Instrument Diagram

DELTA PROCESS MANAGEMENT INC.

DATE 4/20/88 BY CRF
SCALE NONE
DWG. NO. 0-078-PD-06 REV. G



APPROVED
W.R. GRACE & CO. - CONN.

EQ. NO.	CF-201	EQ. NO.	V-207	EQ. NO.	ME-201	EQ. NO.	D-201	EQ. NO.	B-201	EQ. NO.	E-206
TYPE	CENTRIFUGE	TYPE	HOPPER	TYPE	V.B.B.	TYPE	EXCH	TYPE	BLOWER	TYPE	EXCH
NAME	CENTRIFUGE	NAME	DRYER FEED HOPPER	NAME	LIVE BOTTOM BIN	NAME	DRYER	NAME	DRYER EXHAUST BLOWER	NAME	DRYER EXHAUST CONDENSER
VENDOR	◇	VENDOR	◇	VENDOR	◇	VENDOR	◇	VENDOR	◇	VENDOR	◇
MAT'L	304 SS	MAT'L	304 SS	MAT'L	304 SS	MATERIAL	304 SS	MAT'L	304 SS	MATERIAL	304 SS
SIZE	48" x 30"	SIZE	4'D x 4'	SIZE	◇	SIZE	130 S.F.	SIZE	◇	SIZE	100 S.F.
HP	N/A	HP	N/A	HP	N/A	HP	N/A	HP	0	HP	N/A
RPM	N/A	RPM	N/A	RPM	N/A	RPM	N/A	RPM	0	RPM	N/A
CAPACITY	0	CAPACITY	45 CU. FT.	CAPACITY	0	MM BTUH	1.3 MM BTUH	CAPACITY	125 CFM	MM BTUH	0000
TEMP	10 C	TEMP	AMB.	TEMP	AMB.	TEMP	0	TEMP	65 C	TEMP	0
PRES.	ATM	PRES.	ATM	PRES.	ATM	PRES.	0	PRES.	0	PRES.	0

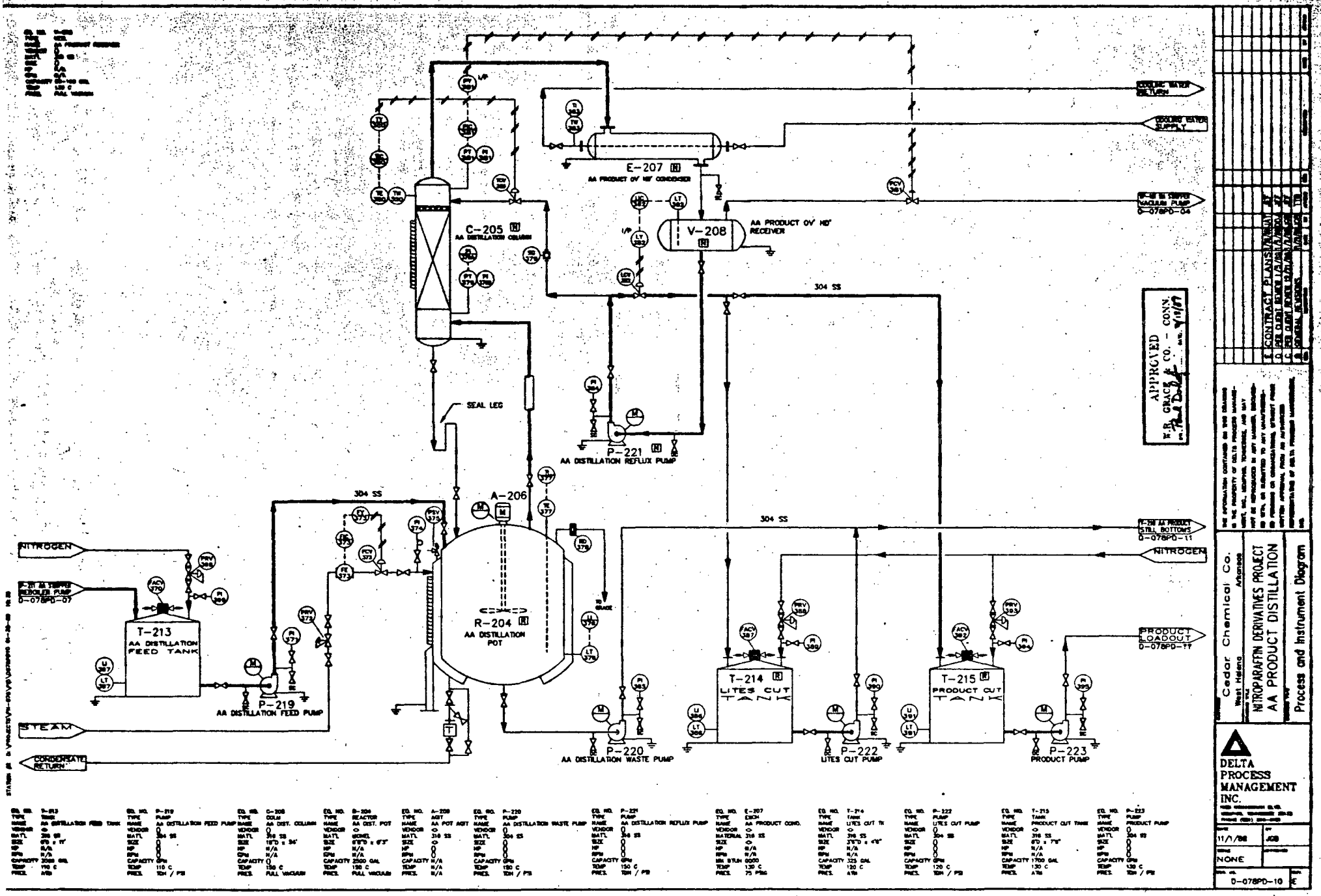
Cedar Chemical Co.
West Helena, Arkansas

NITROPARAFFIN DERIVATIVES PROJECTS
CENTRIFUGATION, DRYING AND PACKAGING

Process and Instrument Diagram

DELTA PROCESS MANAGEMENT INC.

DATE: 4-20-88 | BY: arf
SCALE: NONE
DWG. NO.: D-078PD-08 | REV.: G



Cedar Chemical Co.
West Helena
Arkansas

NITROPARAFFIN DERIVATIVES PROJECT
AA PRODUCT DISTILLATION

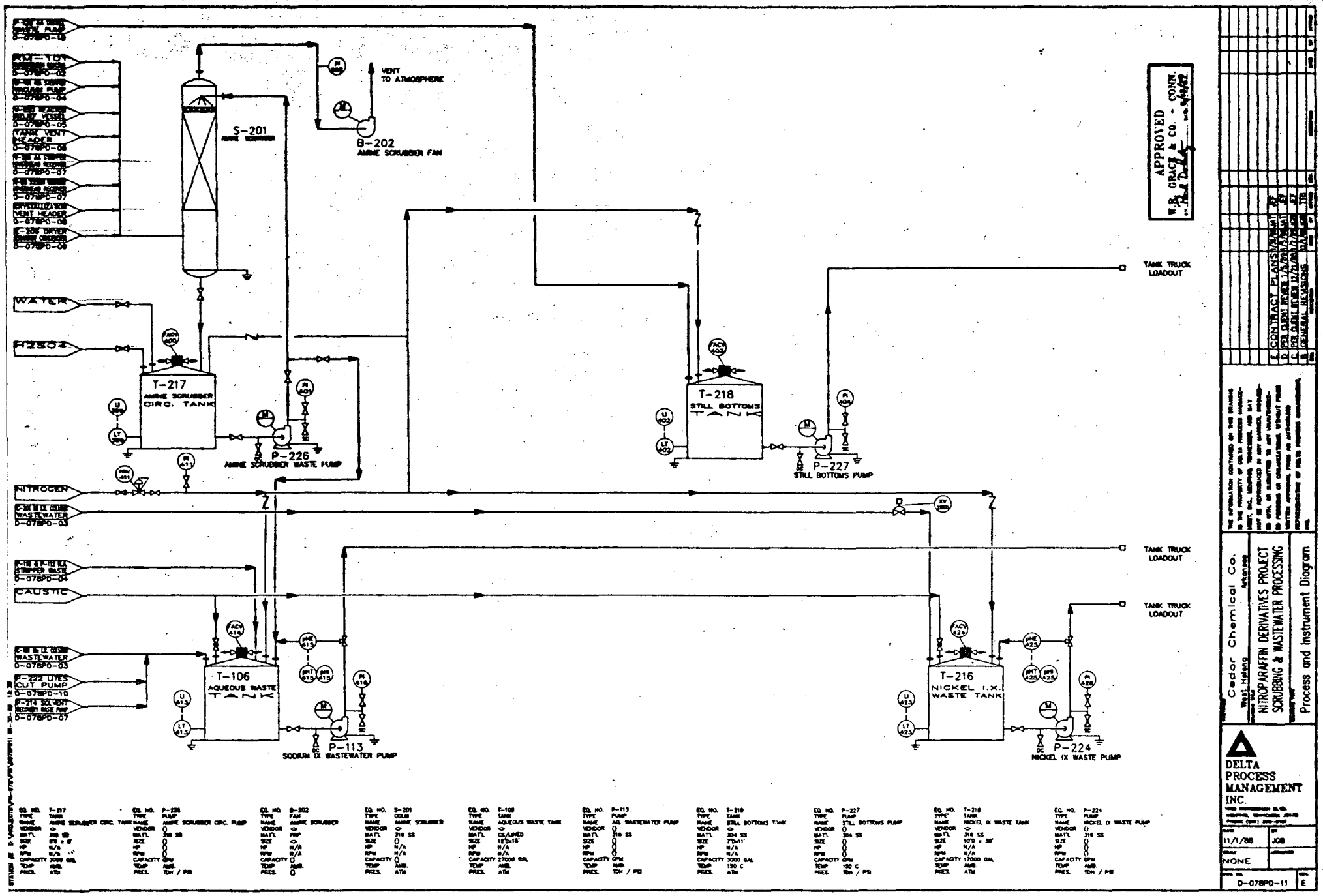
Process and Instrument Diagram

DELTA PROCESS MANAGEMENT INC.

11/1/88

NONE

D-078PD-10



REV	DESCRIPTION	BY	CHK	APP	DATE
F	CONTRACT PLANS				
D	PER DRN REV 1				
C	PER DRN REV 12				
B	GENERAL REVISIONS				

Cedar Chemical Co.
West Helena, Arkansas

NITROPARAFFIN DERIVATIVES PROJECT
SCRUBBING & WASTEWATER PROCESSING

Process and Instrument Diagram

DELTA PROCESS MANAGEMENT INC.

11/1/88	JCB
NONE	
D-078PD-11	E

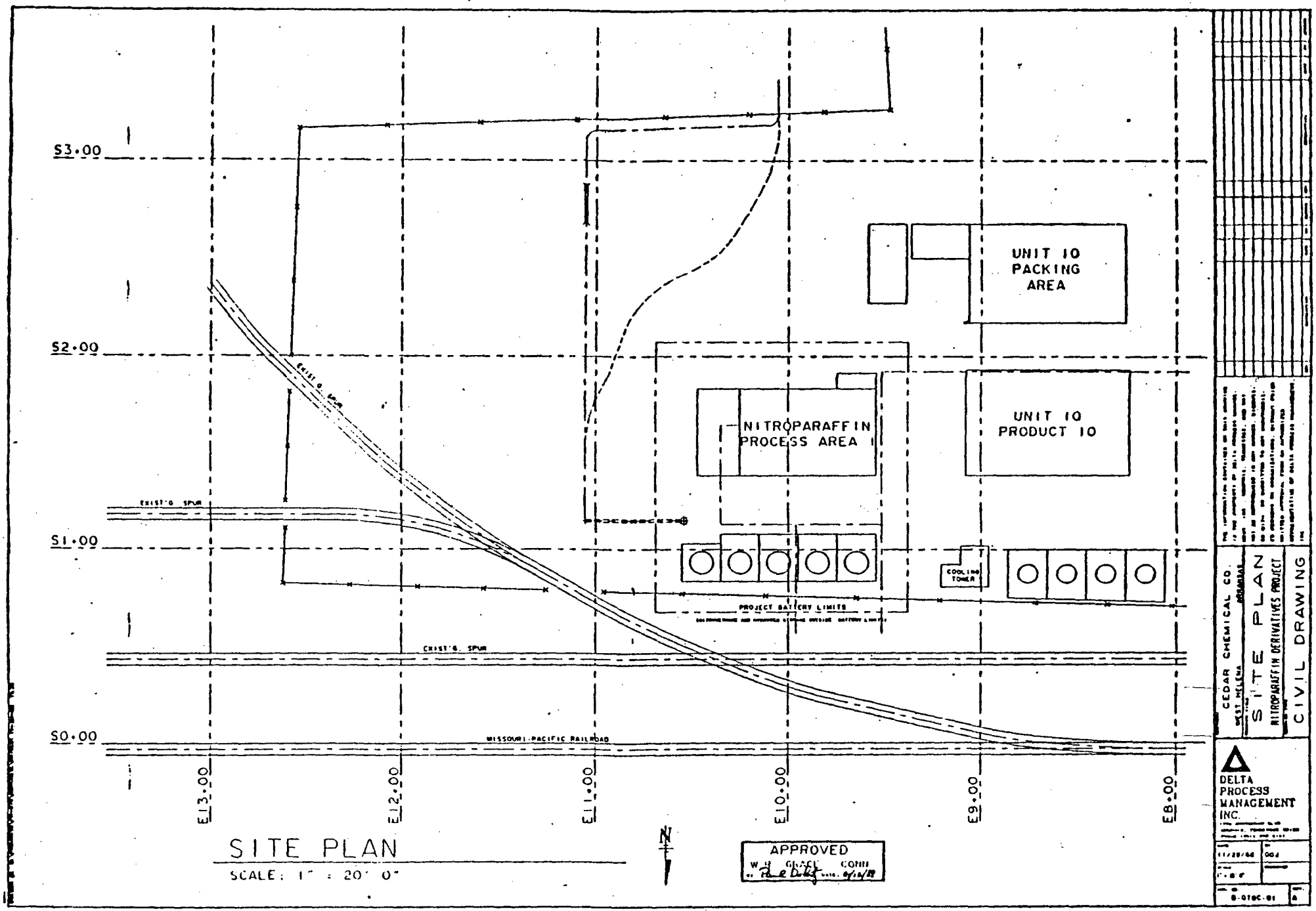

S3+00
S2+00
S1+00
S0+00
E13+00
E12+00
E11+00
E10+00
E9+00
E8+00
UNIT 10
PACKING
AREA
NITROPARAFFIN
PROCESS AREA
UNIT 10
PRODUCT 10
COOLING
TOWER
PROJECT BATTERY LIMITS
EXIST'G SPUR
EXIST'G SPUR
MISSOURI-PACIFIC RAILROAD
SITE PLAN
SCALE: 1" = 20' 0"
N
APPROVED
W.R. GRACE CONN.
CEDAR CHEMICAL CO.
WEST HELENA ARKANSAS
SITE PLAN
NITROPARAFFIN DERIVATIVES PROJECT
CIVIL DRAWING
DELTA
PROCESS
MANAGEMENT
INC.

EXHIBIT B

EXHIBIT C-1

TARGET

Specification Limits for TA

Test	Specification	Test Method
Assay (dry basis)	greater than 99.0%	NPDST 1
Melting Point	167°C minimum	NPDST 4
Color (APHA)	less than 20, 20% solution	NPDST 5
Heavy Metals	less than 10 ppm	NPDST 8
Loss on Drying	less than 1%	NPDST 3

EXHIBIT C-2

TARGET

Specification Limits for 2-AB

Test	Specification	Test Method
Assay	greater than 98.0%	NPDST 2
Impurities	NMAB, less than 1%	NPDST 2
	AMP, less than 0.3%	
Color (APHA)	less than 80, neat	NPDST 5
Specific Gravity	0.943 - 0.948	NPDST 7
Water	less than 0.5%	NPDST 6

EXHIBIT C-3

TARGET

Specification Limits for AMP

Test	Specification	Test Method
Assay	greater than 92.0%	NPDST 2
Color (APHA)	less than 20, neat	NPDST 5
Water	less than 5%	NPDST 6

EXHIBIT D-1

PROCESS DESCRIPTION - TN

1. TN (Tris(hydroxymethyl)nitromethane)

TN is produced by a condensation reaction between CH_2O and NC_1 under carefully controlled conditions of pH and temperature. NaOH is employed to adjust the pH. Following the reaction, the Na^+ ions are removed by ion exchange and the solution is fed to a stripper. The solution is stripped to remove H_2O and is either fed to a hydrogenator, for conversion to TA, or to a carbon column, for TN-50.

The process illustrated in Figure 1.

The details of the process are as follows:

1.1 Condensation Reaction

a. The chemistry of the reaction is as follows:

$$CH_3NO_2 + 3CH_2O \xrightarrow[pH = 8.2\text{-}8.5]{35^\circ C} CH_2OH\text{-}C(CH_2OH)_2\text{-}NO_2$$

b. The operation proceeds as follows: A heel of 44% CH_2O is charged to the reactor. The heel charge is limited to the minimum amount which can be recirculated. The pH is adjusted to 8.2 to 8.5 with 10% NaOH. CH_2O (44%) and NC_1 are simultaneously fed over five hours while the reactor is maintained at a pH of 8.2 to 8.5 and a temperature of 35°C. Recirculation through an external heat exchanger is needed to remove the heat of reaction at a sufficient rate. At the conclusion of the simultaneous feed, the reactor contents are maintained at 35°C and a pH of 8.2-8.5 for one hour.

c. The TN Yield is 98%, based on NC_1.

d. The ratio of CH_2O to NC_1 is 3.05. In addition, sufficient CH_2O is fed to completely react the nitroparaffin impurities (i.e. 2 moles CH_2O/mole NC_2, 1 mole CH_2O/mole $2NC_3$).

e. The average NaOH required to maintain the pH is 0.12 wt. % of the batch.

f. The composition of the NC_1 stream is assumed to be 95% NC_1, 3.5% NC_2, 0.5% $2\text{-}NC_3$, 0.1% H_2O and 0.9% others.

g. The material balance of the condensation reactor is presented in Table 1.1

1.2 NA^+ Ion Exchange

a. The TN solution from the condensation reactor is passed through an ion exchange column containing strong acid cation resin; either Rohm & Haas IR-200 (macroreticular) or IR-120 (gel type). The Na^+ level in the solution is reduced to 20 ppm or less and the pH is reduced to 2.5-3.0.

b. The TN solution fed to the ion exchange column has an average of 690 ppm Na^+.

c. The process limit of the column effluent is 20 ppm.

d. The resin is regenerated with a 50% excess of H_2SO_4. The regenerant is fed to the column as a 5% solution.

e. After blowing down the resin with N_2, the liquid hold up on the resin is 50% of the bed volume.

f. At exhaustion, the column is blown down with N_2 and forward washed with 3 bed volumes of H_2O. The forward wash recovers 98% of the TN solution originally held up in the resin.

g. After the forward wash, the column is regenerated by the following sequence of operations:

1. N_2 blowdown
2. H_2O rinse (2 bed volumes)
3. H_2O backwash (2 bed volumes)
4. Regeneration (per 1.2 d)
5. H_2O rinse (8 bed volumes)
6. H_2O backwash (2 bed volumes)
7. N_2 blowdown

h. The material balance for Na^+ ion exchange is presented in Table 1.2

1.3 Nitroalcohol Stripping

a. A continuous stripper is employed to remove excess H_2O (which accompanies CH_2O fed to the reactor) from the TN solution.

b. The TN solution is stripped at 60°C under a mild vacuum.

c. The yield across stripping is 99% for TN.

d. The first batch in the cycle is diluted by the H_2O held up on the ion exchange resin and is fed directly to the stripper. The remaining batches in the cycle are mixed with dilute TN solution from the forward wash of the Na^+ ion exchange column and fed to the stripper.

e. The TN solution fed to the hydrogenator is concentrated to 65%.

f. The material balance for the nitroalcohol stripper is presented in Table 1.3a for TN fed to hydrogenation.

1.4 Waste Streams

Wastes from the production of TN arise from the following:

a. Na^+ Ion Exchange Column Regeneration
(Table 1.2, Stream 112)

b. Nitroalcohol Stripper Overheads
(Table 1.3a, Streams 117 and 117a)
(Table 1.3b, Streams 117 and 117a)

PROCESS DESCRIPTION - TA

2. TA (Tris(hydroxymethyl)aminomethane)

TN is hydrogenated to TA in an autoclave using Raney nickel catalyst in a CH_3OH solution. After removing the catalyst by filtration, the TA solution is fed to an ion exchange column to remove traces of soluble nickel. The TA solution is carbon treated and fed to a crystallizer. In the crystallizer, the TA solution is cooled and fed to a centrifuge where TA wet cake is isolated. The wet cake is fed to a dryer and sold as TA crystal. The mother liquor from the crystallization is stripped. A portion of the stripped mother liquor is recycled to the carbon columns. The remainder is diluted with H_2O and sold as TA-40.

The process is illustrated in Figure 2.

The details of the process are as follows:

2.1 Hydrogenation

a. The chemistry of the reaction is as follows:

$$\begin{array}{l} CH_2OH \\ | \\ CH_2OH\text{-}C\text{-}CH_2OH \\ | \\ NO_2 \end{array} + 3H_2 \xrightarrow[\text{Raney Ni}]{\substack{40^oC \\ 1000\ psig \\ CH_3OH}} \begin{array}{l} CH_2OH \\ | \\ CH_2OH\text{-}C\text{-}CH_2OH \\ | \\ NH_2 \end{array} + 2H2O$$

b. The autoclave is operated as follows: The 1500 gallon reactor is 40% filled with CH_3OH and a slurry of Raney nickel and is pressurized to 1000 psig with H_2. Steam in the jacket heats the batch to 40°C. When the reactor is at the specified pressure and temperature, TN solution is fed to the reactor at the rate of 19.2 lbs. TN/(lb. catalyst hr.). Fluid is circulated through the internal coils in order to remove the heat of reaction and maintain the batch temperature at 40°C. H_2 is supplied to the autoclave to maintain the reactor at 1000 psig. When the reactor contains 1400 gallons, the TN feed is stopped. The reactor is vented to a scrubber to 50 psig and the TA solution is transferred from the autoclave to a catalyst settling tank.

c. The conversion of TN to TA is accomplished with a 95% yield.

d. The vent gas from the reactor is chiefly H_2 with traces of CH_3OH, H_2O, NH_3 and amines. The level of amines depends upon the extent to which the unreacted nitroparaffins were stripped from the TN solution. The vent gas is scrubbed with an H_2SO_4 solution.

e. The material balance for hydrogenation is presented in Table 2.1. The basis is one autoclave reactor batch.

2.2 Catalyst Handling

a. The TA solution from the catalyst settling tank is passed through a catalyst fines filter. The solids in the catalyst settling tank and the filter cake are washed with CH_3OH to remove residual TA solution. The filtered TA solution and the CH_3OH wash are combined in the Ni^{++} ion exchange column feed tank. The residual catalyst in the settling tank is slurried in CH_3OH and recycled to the autoclave catalyst charge tank. In order to maintain the catalyst bed activity, a portion of fresh catalyst equal to the catalyst fines removed is added.

b. The catalyst is pyrophoric, it must be kept moist or isolated from oxygen.

c. The catalyst residue and filter cake is 50% solids.

d. The CH_3OH wash is three times the residue and filter cake volume and recovers 90% of the TA solution held up on the residue and filter cake.

e. The catalyst slurry from the residue and filter is 25 wt. % solids in CH_3OH.

f. The material balance for catalyst handling is presented in Table 2.2. The basis is one autoclave batch.

2.3 Ni^{++} Ion Exchange

a. The filtered TA solution contains soluble Ni^{++} which must be removed from the solution. The Ni^{++} is removed by ion exchange with a weak acid resin; Rohm & Haas IRC-50.

b. The TA solution fed to the column contains an average of 400 ppm Ni^{++} or less.

c. The column must be regenerated when the effluent exceeds 25 ppm Ni^{++}.

d. The resin is regenerated with a 10% excess of H_2SO_4. The regenerant is fed to the column as a 5% solution.

e. The resin swells by 50% upon contact with TA solution. While this phenomena presents a problem at a laboratory scale, it is expected that the column can operate in a standard downflow manner at a commercial scale. Alternatively, the resin can be preswelled by feeding denickled TA in an upflow fashion to the column. The preswelling would occur after regeneration but before the first TA batch is fed.

f. After blowing down the resin with N_2, the liquid hold up on the resin is 50% of the bed volume.

g. At exhaustion, the column is blown down with N_2 and forward washed with 3 bed volumes of H_2O. The forward wash recovers 98% of the TA solution held up on the resin.

h. After the forward wash, the column is regenerated by the following sequence of operations:

1. N_2 blowdown
2. H_2O rinse (2 bed volumes)
3. H_2O Backwash (2 bed volumes)
4. Regeneration (per 2.3d)
5. H_2O Rinse (8 bed volumes)
6. H_2O Backwash (2 bed volumes)
7. N_2 Blowdown

2.4 Carbon Treatment

a. TA solution from the ion exchange column is combined with TA recycled from the stripper. The TA solution is fed to two carbon columns in series at a flowrate of 1GPM/ft.^{2}. The columns are 4'x8' and contain Calgon APA 12x40 granular.

b. The yield across carbon treatment is 99.9%.

c. The TA solution must be kept warm (60°C) in the carbon column feed tank in order to prevent the crystallization of TA, which will occur at 45°C.

d. The Carbon consumption rate has not been determined.

e. The material balance for carbon treatment is presented in Table 2.4.

2.5 Crystallization

a. The carbon treated TA solution is fed to a crystallizer and cooled. The resulting crystal slurry

is centrifuged and the wet cake is sent to a dryer. The mother liquor is sent to the aminoalcohol stripper feed tank.

b. The crystallizer is a 5000 gallon 316 ss vessel with a working volume of about 4000 gallons.

c. The centrifuge wet cake is about 94% solids.

d. Seed crystals are added to the batch at 0.2 wt. % of the total TA fed to the crystallizer.

e. The material balance for the crystallizer is presented in Table 2.5.

2.6 Drying

a. The wet cakes from the crystallizer are fed to a batch indirect dryer.

b. The crystals are dried at 100°C.

c. The yield across drying is 99%.

d. The dry crystal contains 0.5% moisture.

e. The dryer material balance is presented in Table 2.6.

2.7 Aminoalcohol Stripping

a. The mother liquor from the crystallizer is concentrated in a continuous stripper to a 46-49% solution, with a crystallization temperature of about 70°C.

b. The stripper operates at 100°C and atmospheric pressure.

c. The TA yield across stripping is 99%.

d. The first batch after Ni^{++} column regeneration is fed directly to the stripper because it is diluted with H_2O from the ion exchange column. The remaining batches before the next regeneration are mixed with dilute TA solution (from the forward wash of the Ni^{++} column) before being fed to the stripper.

e. The material balance for the aminoalcohol stripper is presented in Table 2.7.

2.8 Recycle and TA Purge

a. The concentrated TA solution from the stripper is divided; a portion is recycled to the aminoalcohol carbon column and the remainder is purged.

b. On average, about 80% of the stripped TA solution is recycled to the aminoalcohol carbon column.

c. The recycle and purge material balance is presented in Table 2.8.

2.9 Solvent Recovery

a. The aminoalcohol stripper overheads are fed to a batch distillation column to recover CH_3OH for recycle.

b. CH_3OH is recovered at a purity of 98.5% and a yield of 95%.

c. The column operates at 65-70°C and essentially atmospheric pressure.

d. The solvent recovery material balance is presented in Table 2.9.

2.10 Waste Streams

Wastes from the production of TA arise from the following:

a. Ni^{++} Ion Exchange Column Regeneration (Table 2.3, Stream 223).

b. Solvent Recovery Bottoms (Table 2.9, Stream 503).

c. Dryer Exhaust (Table 2.6, Stream 130).

d. Hydrogenator Vent Scrubber ($(NH_4)_2SO_4$ and amine sulfate sludge).

e. Spent Catalyst (Table 2.2, Stream 214).

f. Spent Catalyst Rinses (H_2O, trace organics).

g. Fresh Catalyst Rinses (H_2O, Al_2O_3).

h. Catalyst Filter Media.

i. Spent Carbon.

j. Spent Carbon Rinse Water.

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STREAM NO.		102A	102B	104	101	106
		HEEL	CH2O	NCl	NaOH	DISCHARGE
DESCRIPTION		TO REACTOR	TO REACTOR	TO REACTOR	TO REACTOR	FROM REACTOR
COMPONENTS:	MOL. WT.					
CH2O	30.00	685.02	9766.59			155.69
H2O	18.00	840.71	11988.37	7.01	336.28	13170.18
CH3OH	32.00	31.14	443.94			475.07
NCl	61.00			6940.00		69.40
NC2	75.00			49.37		0.61
2-NCl	89.10			7.01		0.09
NaOH	40.10				37.36	37.36
TN	151.10					16846.91
NMPD	135.10					86.19
NMP	119.10					9.14
H2SO4	98.00					
Na2SO4	142.20					
TA	121.10					
AMPD	105.10					
AMP	89.10					
RANEY Ni	-----					
H2	2.00					
NH3	17.00					
NiSO4	154.70					
Ca(OH)2	74.10					
CaSO4	176.20					
Ni(OH)2	92.70					
(NH4)2SO4	132.00					
OTHER	-----			7.01		286.69
TOTAL lb/batch		1556.87	22196.80	7010.10	373.65	31137.42
VOLUME(gal)		166.69	2376.53	738.44	40.24	3000.69
TEMP (deg C)		35.00	35.00	20.00	20.00	35.00
PRES (mm Hg)		760.00	760.00	760.00	760.00	760.00
DENS (lb/gal)		9.34	9.34	9.49	9.29	10.38

CONDENSATION REACTOR

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Na ION EXCHANGE
30 CU FT RESIN: 4 BATCH /CYCLE

STREAM NO.		110A	110B	111	112	113	114	116	115	117	118	119
		EXHAUSTION						REGENERATION				
DESCRIPTION		B/N 1 FROM I.E. COLUMN	B/N 2-4 FROM I.E. COLUMN	FORWARD WASH TO COLUMN	FORWARD WASH FROM COLUMN	RINSE #1 TO COLUMN	BACKWASH #1 TO COLUMN	H2SO4 TO COLUMN	H2O TO DILUTE ACID	RINSE #2 TO COLUMN	BACKWASH #2 TO COLUMN	TOTAL WASTE STREAM
COMPONENTS:	MOL. WT.											
CH2O	30.00	149.86	155.53		5.52							3.31
H2O	18.00	13530.40	13187.05	5615.24	5177.14	3743.49	3743.49	22.74	5716.88	14973.97	3743.49	31908.51
CH3OH	32.00	457.31	475.07		16.83							9.84
NCl	61.00	66.80	69.40		2.46							1.44
NCl2	75.00	0.53	0.61		0.02							0.00
2-NCl	89.10	0.05	0.09		0.00							0.00
NaOH	40.10											
TN	151.10	16216.89	16946.91		596.93							33.19
NMPD	135.10	82.96	86.18		3.05							0.17
NMP	119.10	8.79	9.14		0.32							0.02
H2SO4	98.00							302.09				119.45
Na2SO4	142.20											265.00
TA	121.10											
AMPD	105.10											
AMP	89.10											
RANEY Ni	-----											
H2	2.00											
NH3	17.00											
NiSO4	154.70											
Ca(OH)2	74.10											
CaSO4	176.20											
Ni(OH)2	92.70											
(NH4)2SO4	132.00											
OTHER	-----	275.97	286.69		10.16							0.66
TOTAL lb/batch		30889.67	31116.83	5615.24	5812.34	3743.49	3743.49	324.82	5716.38	14973.97	3743.49	32358.57
VOLUME(gal)		2997.46	3001.60	673.29	666.22	448.86	448.86	21.93	685.48	1795.44	448.86	3813.40
TEMP (deg C)		30.00	30.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00
PRES (mm Hg)		760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00
DENS (lb/gal)		10.31	10.37	8.34	8.72	8.34	8.34	14.81	8.34	8.34	8.34	8.49

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NITROALCOHOL STRIPPER

TN STRIPPED FOR HYDROGENATION

STREAM NO.		110A	122A	123A	110	120	121	122	123
		B/N 1	B/N 1 NA	B/N 1 NA	B/N 2-4	DILUTE TN	FEED	NA	NA
DESCRIPTION		TO NA	STRIPPER	STRIPPER	TO FEED	TO FEED	TO NA	STRIPPER	STRIPPER
		STRIPPER	OVERHEAD	BOTTOMS	TANK	TANK	STRIPPER	OVERHEAD	BOTTOMS
COMPONENTS:	MOL. WT.								
CH2O	30.00	149.36	18.73	131.13	155.63	1.84	157.50	19.69	137.80
H2O	18.00	13639.40	5460.16	8178.24	13187.05	1725.71	14912.77	6551.92	8360.85
CH3OH	32.00	457.31	456.85	0.46	475.07	5.61	480.68	480.20	0.48
NC1	61.00	66.80	66.14	0.67	69.40	0.82	70.22	69.52	0.71
NC2	75.00	0.59	0.58	0.01	0.61	0.01	0.62	0.61	0.01
2-NC3	89.10	0.08	0.08	0.00	0.09	0.00	0.09	0.09	0.00
NaOH	40.10								
TN	151.10	16216.89	162.17	16054.72	16846.91	198.94	17045.85	170.46	16875.39
NMPD	135.10	83.96	0.83	83.13	86.18	1.02	87.20	0.87	86.33
NMP	119.10	8.79	0.09	8.71	9.14	0.11	9.24	0.09	9.15
H2SO4	98.00								
Na2SO4	142.20								
TA	121.10								
AMPD	105.10								
AMP	89.10								
RANEY Ni	-----								
H2	2.00								
NH3	17.00								
NiSO4	154.70								
Ca(OH)2	74.10								
CaSO4	176.20								
Ni(OH)2	92.70								
(NH4)2SO4	132.00								
OTHER	-----	275.97		275.97	286.69	3.39	290.08		290.08
TOTAL lb/batch		30889.67	6157.64	24732.04	31116.83	1937.45	33054.28	7303.15	25751.13
VOLUME(gal)		2997.46	740.27	2237.66	3001.60	222.07	3218.38	877.72	2397.22
TEMP (deg C)		30.00	60.00	60.00	30.00	25.00	30.00	60.00	60.00
PRES (mm Hg)		760.00	140.00	140.00	760.00	760.00	760.00	140.00	140.00
DENS (lb/gal)		10.31	8.32	10.76	10.37	8.72	10.27	8.32	10.73

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HYDROGENATOR
BATCH PER COND BATCH = 2.76

STREAM NO. / DESCRIPTION		123 AVERAGE TN FED TO BUSS REACTOR	201 CH3OH TO REAC	202 CATALYST TO REAC	203 CATALYST LINE RINSE	204 TN TO REAC	205 HYDROGEN TO REAC	206 VENT FROM REAC	207 DISCHARGE FROM REAC
COMPONENTS:	MOL. WT.								
CH2O	30.00	136.16				16.94			
H2O	18.00	6307.92	0.00	127.09	0.99	2396.15			4676.89
CH3OH	32.00	0.47	0.00	2214.06	65.30	0.17			2038.30
NO1	61.00	0.69				0.25			
NO2	75.00	0.31				0.00			
2-NO3	89.10	0.00				0.00			
NaOH	40.10								
TN	151.10	16670.23				5991.85			
NMPD	135.10	85.28				30.65			
NMP	119.10	9.04				3.25			
H2SO4	98.00								
Na2SO4	142.20								
TA	121.10			23.02					4583.36
AMPD	105.10			0.11					22.76
AMP	89.10			0.01					2.32
RANEY Ni	-----			700.70					700.70
H2	2.00						264.66	4.36	
NH3	17.00							0.44	
NiSO4	154.70								
Ca(OH)2	74.10								
CaSO4	176.20								
Ni(OH)2	92.70								
(NH4)2SO4	132.00								
OTHER	-----	286.55		1.31		103.00		0.25	261.79
TOTAL lb/batch		25496.35	0.00	3066.32	66.29	9164.26	264.66	5.04	12556.49
VOLUME(gal)		2364.83	0.00	359.10	10.04	850.00			1330.69
TEMP (deg C)		60.00	25.00	25.00	20.00	25.00			40.00
PRES (mm Hg)		760.00	760.00	760.00	760.00	760.00			
DENS (lb/gal)		10.78	6.60	8.54	6.60	10.78			9.44

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BUSS BATCH PER COND — CATALYST BATCH = — HANDLING 2.78 — SYSTEM

STREAM NO.		208	209	210	211	212	213	214	215
DESCRIPTION		TA FROM DECANT TANK	CH3OH WASH TO TANK	WASH FROM TANK	TOTAL TA FROM TANK	CH3OH FOR SLURRY	SLURRY FROM FILTER	SPENT CATALYST REMOVED	FRESH CATALYST ADDED
COMPONENTS:	MOL. WT.								
CH2O	30.00								
H2O	18.00	4415.12	14.09	243.68	4658.79	32.01	63.36	6.34	70.07
CH3OH	32.00	2166.05	924.92	695.79	2875.84	2102.09	2403.07	240.01	
NC1	61.00								
NC2	75.00								
2-NC3	89.10								
NaOH	40.10								
TN	151.10								
NMPD	135.10								
NMP	119.10								
H2SO4	98.00								
Na2SO4	142.20								
TA	121.10	4328.16		230.22	4558.39		25.58	2.56	
AMPD	105.10	21.49		1.14	22.63		0.13	0.01	
AMP	89.10	2.19		0.12	2.31		0.01	0.00	
RANEY Ni	-----						700.70	70.07	70.07
H2	2.00								
NH3	17.00								
NiSO4	154.70								
Ca(OH)2	74.10								
CaSO4	176.20								
Ni(OH)2	92.70								
(NH4)2SO4	132.00								
OTHER	-----	247.18		13.15	260.33		1.46	0.15	
TOTAL lb/batch		11194.20	939.01	1184.09	12378.29	2134.11	3251.31	325.13	140.14
VOLUME(gal)		1229.15	142.27	150.38	1377.01	323.35	383.35	38.34	12.01
TEMP (deg C)		40.00	25.00	25.00	40.00	25.00	25.00	25.00	25.00
PRES (mm Hg)		760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00
DENS (lb/gal)		9.11	6.60	7.87	8.99	6.60	8.48	8.48	11.67

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		----- NI ION EXCHANGE -----											
		50 CU FT RESIN: 6 BATCH /CYCLE											
		2.78 ----- EXHAUSTION -----								REGENERATION -----			
STREAM NO.		x 211	220A	220	221	222	223	224	226	225	227	228	22.
DESCRIPTION		TA FED TO COLUMN	B/N 1 FROM I.E. COLUMN	B/N 2-6 FROM I.E. COLUMN	FORWARD WASH TO COLUMN	FORWARD WASH FROM COLUMN	RINSE #1 TO COLUMN	BACKWASH #1 TO COLUMN	H2SO4 TO COLUMN	H2O TO DILUTE ACID	RINSE #2 TO COLUMN	BACKWASH #2 TO COLUMN	TOT. WAST STR
COMPONENTS:	MOL. WT.												
[illegible]	[illegible]												
H2O	18.0	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]		[illegible]							[illegible]
[illegible]	[illegible]												
[illegible]	75.0												
[illegible]	[illegible]												
NaOH	40.10												
TN	151.10												
[illegible]	135.1												
NMP	[illegible]												
H2SO4	98.00								369.22				[illegible]
Na2SO4	142.20												
TA	121.10	12682.11	11753.43	12682.11		310.10							[illegible]
AMPO	105.10	62.97	58.36	62.97		4.52							[illegible]
AMP	89.10	6.42	5.95	6.42		0.46							[illegible]
RANEY Ni	-----												
H2	2.00												
NH3	17.00												
NiSO4	154.70												360
Ca(OH)2	74.10												
CaSO4	176.20												
Ni(OH)2	92.70												
(NH4)2SO4	132.00												
OTHER	-----	724.27	654.92	707.05		51.98							[illegible]
TOTAL lb/batch		34438.27	33469.58	34431.61	14039.10	14169.30	6239.15	6239.15	397.01	6987.23	24956.62	6239.15	51941.
VOLUME(gal)		3831.05	3736.17	3830.61	1683.23	1672.26	748.10	748.10	26.80	837.81	2992.40	748.10	[illegible]
TEMP (deg C)		45.00	40.00	40.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00	[illegible]
PRES (mm Hg)		760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.
DENS (lb/gal)		8.99	8.96	8.99	8.34	8.45	8.34	8.34	14.81	8.34	8.34	8.34	[illegible]

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AA CARBON COLUMN STREAMS
NORMAL PROCESSING

STREAM NO.		220A	234	240A	241A	220	234	240	241
DESCRIPTION		B/N 1 FROM I.E. COLUMN	RECYCLE FROM STRIPPER	B/N 1 TO AA CARB COL	B/N 1 FROM AA CARB COL	B/N 2-6 FROM I.E. COLUMN	RECYCLE FROM STRIPPER	B/N 2-6 TO AA CARB COL	B/N 2-6 FROM AA CARB COL
COMPONENTS:	MOL. WT.								
CH3O	30.00								
H2O	18.00	10582.68	3207.44	16790.12	16790.12	12970.00	3207.44	16178.47	16178.47
CH3OH	32.00	7415.13	80.10	7495.24	7495.24	8001.00	80.10	8081.10	8081.10
NCl	61.00								
NCl	75.00								
2-NCl	89.10								
NaOH	40.10								
TN	161.10								
NMPD	135.19								
NMP	119.10								
H2SO4	98.00								
Na2SO4	142.20								
TA	121.10	11753.43	6414.80	18168.23	18168.23	12682.11	6414.80	19096.91	19096.97
AMPD	105.10	58.36	188.89	247.25	247.25	62.97	188.89	251.86	251.86
AMP	89.10	5.95	24.81	30.76	30.76	6.42	24.81	31.23	31.23
RANEY Ni	-----								
H2	2.00								
NH3	17.00								
NiSO4	154.70								
Ca(OH)2	74.10								
CaSO4	176.20								
Ni(OH)2	92.70								
(NH4)2SO4	132.00								
OTHER	-----	654.02	2264.72	2918.74	2889.55	707.05	2264.72	2971.78	2942.06
TOTAL lb/batch		33469.58	12180.77	45650.35	45617.95	34431.61	12180.77	46612.38	46582.73
VOLUME(gal)		3736.17	1204.93	4927.00	4922.40	3830.61	1204.93	5022.14	5019.19
TEMP (deg C)		40.00	100.00	60.00	60.00	40.00	100.00	60.00	60.00
PRES (mm Hg)		760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00
DENS (lb/gal)		8.96	10.11	9.27	9.27	8.99	10.11	9.28	9.28

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TN & TA
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		CRYSTALLIZER									
STREAM NO.		241A	141A	150	151A	152A	241	141	150	151	152
DESCRIPTION		B/N 1 FEED TO XTAL	B/N 1 SEED TO XTAL	CH3OH CAKE WASH	B/N 1 WET CAKE	B/N 1 MOTHER LIQUOR	FEED TO XTAL	B/N 2-6 SEED TO XTAL	CH3OH CAKE WASH	B/N 2-6 WET CAKE	B/N 2-6 MOTHER LIQUOR
COMPONENTS:	MOL. WT.										
CH2O	30.00										
H2O	18.00	16790.12	0.19	24.62	433.75	16381.18	16173.47	[illegible]	26.78	444.23	[illegible]
CH3OH	32.00	7495.24		1616.71	193.62	8918.30	8081.13		1754.83	221.91	[illegible]
NC1	61.00										
NC2	75.00										
2-NC3	89.10										
NaOH	40.10										
TN	151.10										
NMPD	135.10										
NMP	119.10										
H2SO4	98.00										
Na2SO4	142.20										
TA	121.10	18168.23	36.34		10614.74	7589.83	19096.97	33.19		11522.06	7612.96
AMPD	105.10	247.25	0.07		21.77	225.55	251.86	0.08		23.63	228.31
AMP	89.10	30.76	0.00		1.09	29.68	31.23	0.00		1.14	30.06
RANEY Ni	-----										
H2	2.00										
NH3	17.00										
NiSO4	154.70										
Ca(OH)2	74.10										
CaSO4	176.20										
Ni(OH)2	92.70										
(NH4)2SO4	132.00										
OTHER	-----	2889.55	0.63		185.08	2701.92	2942.06	0.67		200.90	2741.99
TOTAL lb/batch		45621.16	37.23	1541.33	11450.05	35846.46	46582.73	39.14	1781.62	12413.87	35989.52
VOLUME(gal)		4923.40		248.69		4156.55	5019.19		269.94		4186.22
TEMP (deg C)		60.00	25.00	10.00	10.00	10.00	60.00	25.00	10.00	10.00	10.00
PRES (mm Hg)		760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00
DENS (lb/gal)		9.27		6.60		8.62	9.28		6.60		8.59

DATE:12/14/89
TN & TA
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		DRYER			
STREAM NO.		161A B/N 1 DRYER VAPORS	160A B/N 1 DRY PRODUCT	161 B/N 2-6 DRYER VAPORS	160 B/N 2-6 DRY PRODUCT
DESCRIPTION					
COMPONENTS:	MOL. WT.				
CH2O	30.00				
HCO	13.00	380.94	52.81	336.97	57.02
CH3OH	32.06	193.63		221.91	
HCl	61.00				
NCl	75.00				
2-NO3	89.10				
NaOH	40.10				
TN	151.10				
NMPD	135.10				
NMP	119.10				
H2SO4	98.00				
Na2SO4	142.20				
TA	121.10	106.15	10508.60	115.22	11406.84
AMPD	105.10	0.22	21.56	0.24	23.40
AMP	89.10	0.01	1.08	0.01	1.17
RANEY Ni	-----				
H2	2.00				
NH3	17.00				
NiSO4	154.70				
Ca(OH)2	74.10				
CaSO4	176.20				
Ni(OH)2	92.70				
(NH4)2SO4	132.00				
OTHER	-----	1.85	183.23	2.01	198.89
TOTAL lb/batch		682.79	10767.26	726.35	11687.62
VOLUME(gal)		82.46		88.03	
TEMP (deg C)		25.00	100.00	25.00	100.00
PRES (mm Hg)		760.00	760.00	760.00	760.00
DENS (lb/gal)		8.28		8.25	

DATE:12/14/88
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AMINOALCOHOL STRIPPER

STREAM NO.		152 AVERAGE FEED	152A	232A	233A	152	230	231	232	233
DESCRIPTION		TO AA STRIPPER	B/N 1 TO AA STRIPPER	B/N 1 AA STRIPPER OVERHEAD	B/N 1 AA STRIPPER BOTTOMS	B/N 2-6 TO FEED TANK	DILUTE TA TO FEED TANK	FEED TO AA STRIPPER	AA STRIPPER OVERHEAD	AA STRIPPER BOTTOMS
COMPONENTS:	MOL. WT.									
CH2O	30.03									
H2O	18.00	15865.28	16361.18	15824.25	2756.84	15762.10	2825.71	18587.81	14423.14	1858.67
CH3OH	32.00	9499.22	9918.30	9828.11	69.15	9814.23	114.84	9729.04	9661.75	67.29
HCl	61.00									
HCl	75.00									
2-HCl	89.10									
NaOH	40.10									
TN	151.10									
NMPD	135.10									
NMP	119.10									
H2SO4	98.09									
Na2SO4	142.20									
TA	121.10	7609.02	7589.93	75.95	7513.87	7612.86	182.32	7794.88	77.25	7716.34
AMPD	105.19	227.85	225.55	2.25	223.30	225.31	0.99	229.21	2.31	226.89
AMP	89.10	29.99	29.68	0.30	29.39	30.06	0.09	30.15	0.33	29.82
RANEY Ni	-----									
H2	2.00									
NH3	17.00									
NiSO4	154.70									
Ca(OH)2	74.10									
CaSO4	176.20									
Ni(OH)2	92.70									
(NH4)2SO4	132.00									
OTHER	-----	2735.31	2701.92	27.02	2674.90	2741.99	10.40	2752.39	27.52	2724.86
TOTAL lb/batch		35965.68	35846.46	21558.98	14287.57	35989.52	2833.96	38823.48	24169.21	14654.27
VOLUME(gal)		4182.94	4156.55	2822.09	1413.21	4188.23	335.25	4523.40	3156.28	1449.63
TEMP (deg C)		5.00	5.00	100.00	100.00	5.00	25.00	6.46	100.00	100.00
PRES (mm Hg)		760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00
DENS (lb/gal)		8.60	8.62	7.64	10.11	8.59	8.45	8.58	7.66	10.11

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--------- RECYCLE AND TA-40 PURGE ---------

STREAM NO.		233	234	235	236	237
DESCRIPTION		AVERAGE TA FROM STRIPPER	RECYCLE TO CASE COLUMN	PURGE	DILUTION H2O TO PURGE	TA-40 TO STORAGE
COMPONENTS:	MOL. WT.					
CHCO	30.00					
H2O	18.00	3841.66	3307.44	534.11	1479.58	2113.69
CH3OH	32.00	95.94	80.10	15.84		15.84
MC1	61.30					
MC2	75.00					
2-MC3	93.10					
NaOH	40.10					
TN	151.10					
NMPD	135.10					
NMP	119.10					
H2SO4	98.00					
Na2SO4	142.20					
TA	121.10	7683.09	6414.80	1268.21		1268.21
AMPD	105.10	226.29	188.89	37.35		37.35
AMP	89.10	29.75	24.81	4.91		4.91
RANEY Ni	-----					
H2	2.00					
NH3	17.00					
NiSO4	154.70					
Ca(OH)2	74.10					
CaSO4	176.20					
Ni(OH)2	92.70					
(NH4)2SO4	132.00					
OTHER	-----	2716.54	2264.72	448.41		448.41
TOTAL lb/batch		14593.15	12180.77	2408.82	1479.58	3888.40
VOLUME(gal)		1443.56	1204.93	238.28	177.41	412.06
TEMP (deg C)		100.00	100.00	100.00	20.00	20.00
PRES (mm Hg)		760.00	760.00	760.00	760.00	760.00
DENS (lb/gal)		10.11	10.11	10.11	8.34	9.44

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		SOLVENT RECOVERY		
STREAM NO.		501	502	503
DESCRIPTION		AVERAGE FEED TO COLUMN	RECOVERED CH3OH	COLUMN BOTTOMS
COMPONENTS:	MOL. WT.			
CH2O	30.00			
H2O	18.00	14128.49	137.41	13991.09
CH3OH	32.00	9497.97	9023.06	474.91
NC1	61.00			
NC2	75.00			
2-NC3	89.10			
NaOH	40.10			
TN	151.10			
NMPD	135.10			
NMP	119.10			
H2SO4	98.00			
Na2SO4	142.20			
TA	121.10	77.62		77.62
AMPD	105.10	2.31		2.31
AMP	89.10	0.32		0.32
RANEY Ni	-----			
H2	2.00			
NH3	17.00			
NiSO4	154.70			
Ca(OH)2	74.10			
CaSO4	176.20			
Ni(OH)2	92.70			
(NH4)2SO4	132.00			
OTHER	-----	27.44		27.44
TOTAL lb/batch		23734.16	9160.48	14573.67
VOLUME(gal)		3100.58	1382.48	1755.59
TEMP (deg C)		25.00	65.00	65.00
PRES (mm Hg)		760.00	760.00	760.00
DENS (lb/gal)		7.65	6.63	8.30

EXHIBIT D-2

PROCESS DESCRIPTION - 2NB

3. 2NB (2 Nitro-Butanol)

2NB is produced by a condensation reaction between CH_2O and $1\text{-}NC_3$ under carefully controlled conditions of pH and temperature. NaOH is employed to adjust the pH. Following the reaction, the Na^+ ions are removed by ion exchange and the solution is fed to a stripper. The stripped solution is fed to an autoclave for hydrogenation.

The process is illustrated in Figure 3.

The details of the process are as follows:

3.1 Condensation Reaction

a. The chemistry of the reaction is as follows:

$$CH_3CH_2\underset{|}{C}H_2(NO_2) + CH_2O \xrightarrow[pH = 9.5\text{-}10]{25^oC} CH_3CH_2\underset{|}{C}H(NO_2)CH_2OH$$

b. The operation proceeds as follows: A heel of CH_3OH is charged to the reactor. The CH_3OH heel charge is 25 wt. % of the total batch. Before the simultaneous feed begins, 10% of the total $1NC_3$ required is added to the heel. The pH is adjusted to 9.5 to 10.0 with 10% NaOH. CH_2O (44%) and $1NC_3$ are simultaneously fed over two hours while the reactor is maintained at a pH of 9.5 to 10.0 and a temperature of 25°C. Recirculation through an external heat exchanger is needed to remove the heat of reaction at a sufficient rate. At the conclusion of simultaneous feed, the reactor contents are maintained at 25°C for four hours.

c. 97.5% of the CH_2O fed reacts with $1NC_3$; 90% reacts to 2NB and 10% reacts to NEPD.

d. The ratio of CH_2O to nitroparaffins is 0.5.

e. The average amount of NaOH required to maintain the pH is 0.05 wt. % of the batch.

f. The material balance for the condensation reactor is presented in Table 3.1.

3.2 Na^+ Ion Exchange

a. The 2NB solution from the condensation reactor is passed through an ion exchange column containing strong acid cation resin; either Rohm & Haas IR-200

(macroreticular) or IR-120 (gel type). The Na^+ level in the solution is reduced to 20 ppm or less and the pH is reduced to 2.5-3.0.

b. The 2NB solution fed to the ion exchange column has an average of 290 ppm Na^+.

c. The process limit of the column effluent is 20 ppm.

d. The resin is regenerated with a 50% excess of H_2SO_4. The regenerant is fed to the column as a 5% solution.

e. After blowing down the resin with N_2, the liquid hold up on the resin is 50% of the bed volume.

f. At exhaustion, the column is blown down with N_2 and forward washed with 3 bed volumes of H_2O. The forward wash recovers 98% of the 2NB solution originally held up in the resin.

g. After the forward wash, the column is regenerated by the following sequence of operations:

1. N_2 Blowdown
2. H_2O Rinse (2 bed volumes)
3. H_2O Backwash (2 bed volumes)
4. Regeneration (per 3.2 d)
5. H_2O Rinse (8 bed volumes)
6. H_2O Backwash (2 bed volumes)
7. N_2 Blowdown

h. The material balance for Na^+ ion exchange is presented in Table 3.2.

3.3 Nitroalcohol Stripping

a. A continuous stripper is employed to concentrate the 2NB solution before being fed to the hydrogenator.

b. The 2NB solution is stripped at 70°C under a mild vacuum.

c. The yield across stripping is 99% for 2NB.

d. The first batch in the cycle is diluted by the H_2O held up on the ion exchange resin and is fed directly to the stripper. The remaining batches in the cycle are mixed with dilute 2NB solution from the forward wash of the Na^+ ion exchange column and fed to the stripper.

e. The 2NB solution is concentrated to 80%.

f. The material balance for the nitroalcohol stripper is presented in Table 3.3.

3.4 Solvent Recovery

a. The nitroalcohol stripper overheads are fed to a batch distillation column to recover CH_3OH for recycle. The column bottoms undergo a phase split. The NP phase is recycled to the NP plant. The water phase is a waste stream.

b. CH_3OH is recovered at a purity of 98.5% and a yield of 95%.

c. The column operates at 65-70°C and essentially atmospheric pressure.

d. The solvent recovery material balance is presented in Table 3.4.

3.5 Waste Streams

Wastes from the production of 2NB arise from the following:

a. Na^+ Ion Exchange Column Regeneration
(Table 3.4, Stream 113)

b. Solvent Recovery Bottoms
(Table 3.4, Stream 505)

PROCESS DESCRIPTION - 2AB

4. 2AB (2-Amino-Butanol)

2NB is hydrogenated to 2AB in an autoclave over Raney nickel catalyst in a CH_3OH solution. After removing the catalyst by filtration, the 2AB solution is fed to an ion exchange column to remove traces of soluble nickel. The 2AB solution is stripped and stored. When several batches have been accumulated, the 2AB is fed to a batch distillation column for product isolation.

The process is illustrated in Figure 4.

The details of the process are as follows:

4.1 Hydrogenation

a. The chemistry of the reaction is as follows:

$$CH_3CH_2CH(NO_2)CH_2OH + 3H_2 \xrightarrow[\text{Raney Ni}]{\substack{50^\circ C \\ 1000\ psig \\ CH_3OH}} CH_3CH_2CH(NH_2)CH_2OH + 2H_2O$$

b. The autoclave is operated as follows: The 1000 gallon reactor is 40% filled with CH_3OH and a slurry of Raney nickel and is pressurized to 1000 psig with H_2. Steam in the jacket heats the batch to 50°C. When the reactor is at the specified pressure and temperature, 2NB solution is fed to the reactor at the rate of 9.2 lbs. 2NB/(lb. catalyst hr.). Fluid is circulated through the internal coils in order to remove the heat of reaction and maintain the batch temperature at 50°C. H_2 is supplied to the autoclave to maintain the reactor at 1000 psig. When the reactor contains 1450 gallons, the 2NB feed is stopped. The reactor is vented to a scrubber to 50 psig and the 2AB solution is transferred from the autoclave to a catalyst settling tank.

c. The conversion of 2NB to 2AB is accomplished with a 95% yield.

d. The vent gas from the reactor is chiefly H_2 with traces of CH_3OH, H_2O, NH_3 and amines. The level of amines depends upon the extent to which the unreacted nitroparaffins were stripped from the 2NB solution. The vent gas is scrubbed with an H_2SO_4 solution.

e. The feed to the hydrogenator is an 80% 2NB solution.

f. The material balance for hydrogenation is presented in Table 4.1. The basis is one autoclave reactor batch.

4.2 Catalyst Handling

a. The 2AB solution from the catalyst settling tank is passed through a catalyst fines filter. The solids in the catalyst settling and the filter cake are washed with CH_3OH to remove residual 2AB solution. The filtered 2AB solution and the CH_3OH wash are combined in the Ni^{++} ion exchange column feed tank. The residual catalyst in the settling tank is slurried in CH_3OH and recycled to the autoclave catalyst charge tank. In order to maintain the catalyst bed activity, a portion of fresh catalyst equal to the catalyst fines removed is added.

b. The catalyst is pyrophoric, it must be kept moist or isolated from oxygen.

c. The catalyst residue and filter cake are 50% solids.

d. The CH_3OH wash is three times the residue and filter cake volume and recovers 90% of the 2AB solution held up on the residue and filter cake.

e. The catalyst slurry from the residue and filter is 25 wt. % solids in CH_3OH.

f. The material balance for catalyst handling is presented in Table 4.2

4.3 Ni^{++} Ion Exchange

a. The filtered 2AB solution contains soluble Ni^{++} which must be removed from the solution. The Ni^{++} is removed by ion exchange with a weak acid resin, Rohm & Haas IRC-50.

b. The 2AB solution fed to the column contains an average of 400 ppm Ni^{++}. The solution leaving the column must contain 25 ppm Ni^{++} or less.

c. The resin is regenerated with a 10% excess of H_2SO_4. The regenerant is fed to the column as a 5% solution.

d. The resin swells by 50% upon contact with 2AB solution. While this phenomena presents a problem at a laboratory scale, it is expected that the column can operate in a standard downflow manner at a commercial scale. Alternatively, the resin can be preswelled by feeding denickled 2AB in an upflow fashion to the

column. The preswelling would occur after regeneration but before the first 2AB batch is fed.

e. After blowing down the resin with N_2, the liquid hold up on the resin is 50% of the bed volume.

f. At exhaustion, the column is blown down with N_2 and forward washed with 3 bed volumes of H_2O. The forward wash recovers 98% of the 2AB solution held up on the resin.

g. After the forward wash, the column is regenerated by the following sequence of operations:

1. N_2 Blowdown
2. H_2O Rinse (2 bed volumes)
3. H_2O Backwash (2 bed volumes)
4. Regeneration (per 4.3d)
5. H_2O Rinse (8 bed volumes)
6. H_2O Backwash (2 bed volumes)
7. N_2 Blowdown

h. The material balance for Ni^{++} ion exchange is presented in Table 4.3

4.4 Aminoalcohol Stripping

a. The denickled 2AB solution is concentrated in a continuous stripper to a 80% solution.

b. The stripper operates at 100°C and atmospheric pressure.

c. The 2AB yield across stripping is 99%.

d. The first batch after Ni^{++} column regeneration is fed directly to the stripper because it is diluted with H_2O from the ion exchange column. The remaining batches before the next regeneration are mixed with dilute 2AB solution (from the forward wash of the Ni^{++} column) before being fed to the stripper.

e. The material balance for the aminoalcohol stripper is presented in Table 4.4.

4.5 Distillation

a. Several batches of stripped 2AB are accumulated and fed to a batch distillation column. After obtaining a lites cut, a product cut is taken overhead to isolate 2AB. The still bottoms represent a waste stream.

b. The batch distillation column operates at a pot temperature of 150°C.

c. The lites cut is obtained at about 600 mm Hg.

d. The product cut is obtained at about 300 mm Hg.

e. 2AB is recovered at a 95% yield and a purity of 99%.

f. The material balance for batch distillation is presented in Table 4.5.

4.6 Solvent Recovery

a. The aminoalcohol stripper overheads are fed to a batch distillation column to recover CH_3OH for recycle.

b. CH_3OH is recovered at a purity of 98.5% and a yield of 95%.

c. The column operates at 65-70°C and essentially atmospheric pressure.

d. The solvent recovery material balance is presented in Table 4.6.

4.7 Waste Streams

Wastes from the production of 2AB arise from the following:

a. Ni^{++} Ion Exchange Column Regeneration (Table 4.3, Stream 223).

b. Solvent Recovery Bottoms (Table 4.6, Stream 503).

c. Distillation Bottoms (Table 4.5, Stream 404).

d. Hydrogenator Vent Scrubber ($(NH_4)_2SO_4$ and amine sulfate sludge).

e. Spent Catalyst (Table 4.2, Stream 214).

f. Spent Catalyst Rinses (H_2O, trace organics).

g. Fresh Catalyst Rinses (H_2O, Al_2O_3).

h. Catalyst Filter Media.

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2NB & 2AB
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condensation

STREAM NO.		103	104	101	102	106
DESCRIPTION		HEEL TO REACTOR	1-NC3 TO REACTOR	NaOH TO REACTOR	CH2O TO REACTOR	DISCHARGE FROM REACTOR
COMPONENTS:	MOL. WT.					
H2O	18.00				3270.47	26.25
EOH	18.00	93.86	6.74	112.63	2786.49	2999.72
CH3OH	32.00	6163.44			103.20	6256.63
1-NC3	89.10		12082.00			6868.05
2-NC3	89.10		67.43			34.05
2M2NC3	103.00		67.43			67.43
1-NC4	103.00		101.15			48.53
2-NC4	103.00		101.15			51.09
NaOH	40.01			12.51		12.51
2-NB	119.10					7869.02
NEPD	149.10					547.29
NMP	119.10					44.62
2NC5OH	131.00					63.68
2N2P13PD	161.00					3.91
2N2MB	133.00					64.65
H2SO4	98.00					
Na2SO4	142.00					
2-AB	89.10					
AEPD	119.10					
AMP	89.10					
2AC5OH	101.00					
2A2P13PD	131.00					
2A2MB	103.00					
RANEY Ni	-----					
S2	2.00					
NH3	17.03					
NiSO4	154.70					
Ca(OH)2	74.10					
CaSO4	176.20					
Ni(OH)2	92.70					
(NH4)2SO4	132.00					
OTHER	-----		60.33			61.71
TOTAL lb/batch		6257.30	13486.50	125.15	5160.16	25029.81
VOLUME(gal)		948.08	1626.23	13.48	552.27	2995.92
TEMP (deg C)		20.00	20.00	20.00	35.00	25.00
PRES (mm Hg)		760.00	760.00	760.00	760.00	760.00
DENS (lb/gal)		6.60	8.29	9.29	9.34	8.35

DATE:2/23/89
2NB & 2AB
CEDAR
WEST HELENA AR

ION EXCHANGE
30 CU FT RESIN: 10 BATCH /CYCLE

STREAM NO.		110A	110	111	112	113	114	116	115	117	118	119
DESCRIPTION		B/N 1 FROM I.E. COLUMN	B/N 2-10 FROM I.E. COLUMN	FORWARD WASH TO COLUMN	FORWARD WASH FROM COLUMN	RINSE #1 TO COLUMN	BACKWASH #1 TO COLUMN	H2SO4 TO COLUMN	H2O TO DILUTE ACID	RINSE #2 TO COLUMN	BACKWASH #2 TO COLUMN	TOTAL WASTE STREAM
COMPONENTS:	MOL. WT.											
CH2O	30.00	25.27	26.25		0.98							1.05
H2O	18.00	3828.87	3985.35	5615.24	4817.00	3743.49	3743.49	22.74	5716.88	14973.97	3743.49	31916.75
CH3OH	32.00	6021.92	6266.65		222.36							16.38
1-NC3	89.10	6610.80	6856.05		245.70							18.88
2-NC3	89.10	32.78	34.05		1.21							0.37
2M2NC3	103.00	64.91	67.43		2.39							0.13
1-NC4	103.00	46.76	48.58		1.76							0.26
2-NC4	103.00	49.17	51.09		1.85							0.06
NaOH	40.01											
2-NB	119.10	7574.28	7869.02		279.22							15.52
NBPD	149.10	526.79	547.29		19.42							1.08
NMP	119.10	42.95	44.62		1.58							0.09
2NC5OH	131.00	61.29	63.68		2.26							0.13
2N2P13PD	161.00	3.77	3.91		0.14							0.01
2N2MB	133.00	62.23	64.65		2.29							0.13
H2SO4	98.00							302.09				146.82
Na2SO4	142.00											222.05
2-AB	89.10											
ABPD	119.10											
AMP	89.10											
2AC5OH	101.00											
2A2P13PD	131.00											
2A2MB	103.00											
RANEY Ni	-----											
H2	2.00											
NH3	17.00											
NiSO4	154.70											
Ca(OH)2	74.10											
CaSO4	176.20											
Ni(OH)2	92.70											
(NH4)2SO4	132.00											
OTHER	-----	59.40	61.71		2.19							0.15
TOTAL lb/batch		25021.17	25022.32	5615.24	5598.31	3743.49	3743.49	324.82	5716.88	14973.97	3743.49	32333.97
VOLUME(gal)		2996.79	2996.79	673.29	671.14	448.86	448.86	21.93	685.48	1795.44	448.86	3817.67
TEMP (deg C)		25.00	25.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00
PRES (mm Hg)		760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00
DENS (lb/gal)		8.35	8.35	8.34	8.34	8.34	8.34	14.81	8.34	8.34	8.34	8.47

DATE:2/23/89
2NB & 2AB
CEDAR
WEST HELENA AR

		NITROALCOHOL STRIPPER							
STREAM NO.		110A	122A	123A	110	120	121	122	123
DESCRIPTION		B/N 1 TO NA STRIPPER	B/N 1 NA STRIPPER OVERHEAD	B/N 1 NA STRIPPER BOTTOMS	B/N 2-10 TO FEED TANK	DIL. 2NB TO FEED TANK	FEED TO NA STRIPPER	NA STRIPPER OVERHEAD	NA STRIPPER BOTTOMS
COMPONENTS:	MOL. WT.								
CH2O	30.30	25.27	3.16	22.11	26.25	0.10	26.35	3.29	23.06
H2O	18.00	3928.67	2900.75	928.12	3005.35	535.22	3540.57	2618.91	1036.77
CH3OH	32.00	6031.92	6025.89	6.03	6266.65	24.71	6291.36	6285.06	6.29
1-NC3	89.10	6610.80	6544.69	66.11	6868.05	27.08	6895.13	6826.18	68.95
2-NC3	89.10	32.78	32.45	0.33	34.05	0.13	34.19	33.85	0.34
2M2NC3	103.00	64.91	64.26	0.65	67.43	0.27	67.70	67.02	0.68
1-NC4	103.00	46.76	46.29	0.47	48.58	0.20	48.77	48.28	0.49
2-NC4	103.00	49.17	48.68	0.49	51.08	0.21	51.29	50.77	0.51
NaOH	40.01								
2-NB	119.10	7574.28	75.74	7498.54	7869.02	31.02	7900.05	79.00	7821.05
NEPD	149.10	526.79	5.27	521.52	547.29	2.16	549.44	5.49	543.95
NMP	119.10	42.95	0.43	42.52	44.62	0.18	44.79	0.45	44.35
2NC5OH	131.00	61.29	0.61	60.68	63.68	0.25	63.93	0.64	63.29
2N2P13PD	161.00	3.77	0.04	3.73	3.91	0.02	3.93	0.04	3.89
2N2MB	133.00	62.23	0.62	61.61	64.65	0.25	64.91	0.65	64.26
H2SO4	98.00								
Na2SO4	142.00								
2-AB	89.10								
AEPD	119.10								
AMP	89.10								
2AC5OH	101.00								
2A2P13PD	131.00								
2A2MB	103.00								
RANEY Ni	-----								
H2	2.00								
NH3	17.00								
NiSO4	154.70								
Ca(OH)2	74.10								
CaSO4	176.20								
Ni(OH)2	92.70								
(NH4)2SO4	132.00								
OTHER	-----	59.40		59.40	61.71	0.24	61.95		61.95
TOTAL lb/batch		25021.17	15748.88	9272.29	25022.32	622.03	25644.36	15914.54	9723.82
VOLUME(gal)		2996.79	2056.72	973.49	2996.79	74.57	3071.36	2084.33	1022.29
TEMP (deg C)		25.00	70.00	70.00	25.00	25.00	25.00	70.00	70.00
PRES (mm Hg)		760.00	225.00	225.00	760.00	760.00	760.00	225.00	225.00
DENS (lb/gal)		8.35	7.66	9.52	8.35	8.34	8.35	7.64	9.52

DATE:2/23/89
2NB & 2AB
CEDAR
WEST HELENA AR

STREAM NO.		501	502	503	504	505
DESCRIPTION		AVG NA STRIPPER OVERHEAD	RECOVERED CH3OH	COLUMN BOTTOMS	ORGANIC PHASE	AQUEOUS PHASE
COMPONENTS:	MOL. WT.					
CH2O	30.00	3.23		3.23	0.16	3.12
H2O	18.00	2550.59	90.55	2461.95	225.43	2166.52
CH3OH	32.00	6259.14	5946.19	312.96	56.33	256.62
1-NC3	89.10	6798.03		6798.03	6736.85	61.18
2-NC3	89.10	33.71		33.71	33.40	0.30
2M2NC2	103.00	66.75		66.75	66.21	0.53
1-NC4	103.00	48.09		48.09	47.84	0.24
2-NC4	103.00	50.56		50.56	50.31	0.25
NaOH	40.01					
2-NB	119.10	78.67		78.67	61.37	17.31
NEPD	149.10	5.47		5.47	2.57	2.90
NMP	119.10	0.45		0.45	0.30	0.15
2NC5OH	131.00	0.64		0.64	0.43	0.21
2N2P13PD	161.00	0.04		0.04	0.02	0.02
2N2MB	133.00	0.65		0.65	0.43	0.21
H2SO4	98.00					
Na2SO4	142.00					
2-AB	89.10					
ABPD	119.10					
AMP	89.10					
2AC5OH	101.00					
2A2P13PD	131.00					
2A2MB	103.00					
RANEY Ni	-----					
H2	2.00					
NH3	17.00					
NiSO4	154.70					
Ca(OH)2	74.10					
CaSO4	176.20					
Ni(OH)2	92.70					
(NH4)2SO4	132.00					
OTHER	-----	0.00		0.00		
TOTAL lb/batch		15897.97	6036.74	9861.23	7351.66	2509.57
VOLUME(gal)		2081.57	914.66	1194.34	897.44	306.94
TEMP (deg C)		25.00	65.00	70.00	70.00	70.00
PRES (mm Hg)		760.00	760.00	760.00	760.00	760.00
DENS (lb/gal)		7.64	6.60	8.25	8.28	8.18

DATE:2/23/89
2NB & 2AB
CEDAR
WEST HELENA AR

STREAM NO.		123 AVERAGE NA STRIPPER BOTTOMS
DESCRIPTION		
COMPONENTS:	MOL. WT.	
CH2O	30.00	22.36
H2O	18.00	1016.90
CH3OH	32.00	6.27
1-NC3	89.10	68.67
2-NC3	89.10	0.34
2M2NC3	103.00	0.67
1-NC4	103.00	0.43
2-NC4	103.00	0.51
NaOH	40.01	
2-NB	119.10	7788.79
NEPD	149.10	541.71
NMP	119.10	44.16
2NC5OH	131.00	63.03
2N2P13PD	161.00	3.87
2N2MB	133.00	63.99
H2SO4	98.00	
Na2SO4	142.00	
2-AB	89.10	
AEPD	119.10	
AMP	89.10	
2AC5OH	101.00	
2A2P13PD	131.00	
2A2MB	103.00	
RANEY Ni	-----	
H2	2.00	
NH3	17.00	
NiSO4	154.70	
Ca(OH)2	74.10	
CaSO4	176.20	
Ni(OH)2	92.70	
(NH4)2SO4	132.00	
OTHER	-----	61.70
TOTAL lb/batch		9684.07
VOLUME(gal)		1017.41
TEMP (deg C)		70.00
PRES (mm Hg)		760.00
DENS (lb/gal)		9.52

	HYDROGENATOR						
	H2 BATCH PER COND BATCH = 1.20						
	201 CH3OH TO REAC	202 CATALYST TO REAC	203 CATALYST LINE RINSE	204 2-NB TO REAC	205 HYDROGEN TO REAC	206 VENT FROM REAC	207 DISCHARGE FROM REAC
				19.07			
	1.99	91.45	0.99	844.56			3967.09
	123.90	1666.60	65.27	5.20			1260.37
				67.03			
				0.26			
				0.66			
				0.40			
				0.42			
				6468.89			
				449.91			
				36.68			
				52.35			
				3.22			
				53.15			
		20.14					4597.48
		1.50					341.41
		0.11					25.07
		0.17					39.34
		0.01					2.49
		0.17					39.10
		527.62					527.62
					358.93	4.29	
						0.43	
		1.49		51.24		58.70	333.29
	125.79	2309.26	66.26	3042.99	358.93	63.41	10839.36
	18.98	271.02	10.00	845.00			1319.53
	25.00	25.00	25.00	25.00			50.00
	760.00	760.00	760.00	760.00			
	6.63	8.52	6.63	9.52			8.21

DATE:2/23/89
2NB & 2AB
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WEST HELENA AR

CATALYST HANDLING SYSTEM
H2 BATCH PER COND BATCH = 1.20

STREAM NO.		208	209	210	211	212	213	214	215
DESCRIPTION		2AB FROM DECANT TANK	CH3OH WASH TO TANK	WASH FROM TANK	TOTAL 2AB FROM TANK	CH3OH FOR SLURRY	SLURRY FROM TANK	SPENT CATALYST REMOVED	FRESH CATALYST ADDED
COMPONENTS:	MOL. WT.								
CH20	30.00								
H20	18.00	2917.30	10.61	141.01	1058.81	24.10	42.39	4.30	30.79
CH3OH	32.00	1770.39	696.46	518.13	2238.52	1582.87	1851.78	185.15	
1-NC3	89.10								
2-NC3	89.10								
2M2NC3	103.00								
1-NC4	103.00								
2-NC4	103.00								
NaOH	40.01								
2-NB	119.10								
NEPD	149.10								
NMP	119.10								
2NC5OH	131.00								
2N2P13PD	161.00								
2N2MB	133.00								
H2SO4	98.00								
Na2SO4	142.00								
2-AB	89.10	4373.70		201.40	4575.10		22.38	2.24	
ABPD	119.10	324.80		14.96	339.75		1.66	0.17	
AMP	89.10	24.80		1.14	25.94		0.13	0.01	
2AC5OH	101.00	36.48		1.68	38.16		0.19	0.02	
2A2P13PD	131.00	2.37		0.11	2.47		0.01	0.00	
2A2MB	103.00	37.20		1.71	38.91		0.19	0.02	
RANEY Ni	-----						527.62	52.76	52.76
H2	2.00								
NH3	17.00								
NiSO4	154.70								
Ca(OH)2	74.10								
CaSO4	176.20								
Ni(OH)2	92.70								
(NH4)2SO4	132.00								
OTHER	-----	322.78		14.86	337.64		1.65	0.17	
TOTAL lb/batch		9810.30	707.07	895.00	10705.30	1606.97	2448.50	244.86	135.52
VOLUME(gal)		1246.90	106.71	123.40	1369.60	242.52	289.57	28.96	9.04
TEMP (deg C)		25.00	25.00	25.00	25.00	25.00	25.00	25.00	25.00
PRES (mm Hg)		760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00
DENS (lb/gal)		7.87	6.63	7.25	7.82	6.63	8.46	8.46	11.67

\T2:2/22/89
4B & 2AB
3DAB
3ST HELENA AE

Ni ION EXCHANGE 50 CU FT RESIN: 10 BATCH /CYCLE

EAM NO.		211 2AB	220A B/N 1	220 B/N 2-10	221 FORWARD	222 FORWARD	223 RINSE #1	224 BACKWASH	226 H2SO4	235 H2O TO	237 RINSE #2	228 BACKWASH	22 TOT
SCRIPTION		FED TO COLUMN	FROM I.E. COLUMN	FROM I.E. COLUMN	WASH TO COLUMN	WASH FROM COLUMN	TO COLUMN	#1 TO COLUMN	TO COLUMN	DILUTE ACID	TO COLUMN	#2 TO COLUMN	WAS STRE
MPONENTS:	MOL. WT.												
2O	18.00	3682.33	4620.13	3684.90	14003.10	10010.42	9358.73	9358.73	27.79	6987.29	24956.82	6239.15	5760
3OH	32.00	2755.46	2286.70	2755.46		459.39							
-NO3	63.10												
-NO3	89.10												
2NO3	103.00												
-NO4	103.00												
-NO4	103.00												
OH	40.01												
-NB	119.10												
PD	149.10												
P	119.10												
C5OH	131.00												
2P13PD	161.00												
2NB	133.00												
2SO4	98.00								369.22				261
a2SO4	142.00												
-AB	89.10	5508.59	4571.47	5508.59		918.38							18.
PD	119.10	409.07	339.48	409.07		68.20							1.
F	89.10	31.23	25.92	31.23		5.21							0.
C5OH	101.00	45.94	38.13	45.94		7.66							0.
2P13PD	131.00	2.98	2.47	2.98		0.50							0.
2NB	103.00	46.85	38.88	46.85		7.81							0.
NET Ni	-----												
	2.00												
3	17.00												
SO4	154.70												169.
(OH)2	74.10												
SO4	175.20												
(OH)2	92.70												
H4)2SO4	132.00												
HBB	-----	406.53	337.37	400.09		67.78							59.
TAL lb/batch		12889.60	12260.56	12885.13	14038.10	13847.34	9358.73	9358.73	397.01	6987.29	24956.82	6239.15	58157.
LUME(gal)		1649.05	1555.29	1648.69	1683.23	1676.69	1122.15	1122.15	26.80	837.81	2992.40	748.10	6392.
MP (deg C)		25.00	25.00	25.00	25.00	25.00	25.00	25.00	25.00	25.00	25.00	25.00	25.0
ES (mm Hg)		760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.
NS (lb/gal)		7.82	7.88	7.82	8.34	8.26	8.34	8.34	14.81	8.34	8.34	8.34	8.

DATE:2/23/89
2NB & 2AB
CEDAR
WEST HELENA AR

		AMINOALCOHOL STRIPPER							
STREAM NO.		220A	230A	231A	220	230	231	232	233
DESCRIPTION		B/N 1 TO AA STRIPPER	B/N 1 AA STRIPPER OVERHEAD	B/N 1 AA STRIPPER BOTTOMS	B/N 2-10 TO FEED TANK	DIL. 2AB TO FEED TANK	FEED TO AA STRIPPER	AA STRIPPER OVERHEAD	AA STRIPPER BOTTOMS
COMPONENTS:	MOL. WT.								
CH2O	30.00								
H2O	18.00	4620.13	4388.12	231.01	3694.30	1368.05	5062.35	4890.30	172.05
CH3OH	32.00	2286.70	2265.84	20.87	2755.46	51.04	2806.51	2778.44	28.07
1-NC3	89.10								
2-NC3	89.10								
2M2NC3	103.00								
1-NC4	103.00								
2-NC4	103.00								
NaOH	40.01								
2-NB	119.10								
NBPD	149.10								
NMP	119.10								
2NC5OH	131.00								
2N2P13PD	161.00								
2N2MB	133.00								
H2SO4	98.00								
Na2SO4	142.00								
2-AB	89.10	4571.47	45.71	4525.75	5508.59	102.04	5610.64	56.11	5554.53
AEPD	119.10	339.48	3.39	336.09	409.07	7.58	416.65	4.17	412.49
AMP	89.10	25.92	0.26	25.66	31.23	0.58	31.81	0.32	31.49
2AC5OH	101.00	38.13	0.38	37.74	45.94	0.85	46.79	0.47	46.32
2A2P13PD	131.00	2.47	0.02	2.45	2.98	0.06	3.03	0.03	3.00
2A2MB	103.00	38.88	0.39	38.49	46.85	0.87	47.72	0.48	47.24
RANEY Ni	-----								
H2	2.00								
NH3	17.00								
NiSO4	154.70								
Ca(OH)2	74.10								
CaSO4	176.20								
Ni(OH)2	92.70								
(NH4)2SO4	132.00								
OTHER	-----	337.37	33.74	303.64	400.09	7.53	407.62	4.08	403.54
TOTAL lb/batch		12260.56	6736.86	5523.70	12885.13	1538.59	14423.72	7644.39	6779.34
VOLUME(gal)		1555.29	868.11	687.63	1648.69	186.30	1834.46	992.14	843.15
TEMP (deg C)		25.00	100.00	100.00	25.00	25.00	25.00	100.00	100.00
PRES (mm Hg)		760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00
DENS (lb/gal)		7.88	7.76	8.03	7.82	8.26	7.86	7.70	8.04

DATE:2/23/89
2NB & 2AB
CEDAR
WEST HELENA AR

PRODUCT DISTILLATION

STREAM NO.		401	402	404	405
DESCRIPTION		FEED TO DIST	LITES CUT	PRODUCT CUT	DIST BOTTOMS
COMPONENTS:	MOL. WT.				
CH2O	30.00				
H2O	18.00	259.48	232.28	18.69	8.50
CH3OH	32.00	27.55	27.55		
1-NC3	89.10				
2-NC3	89.10				
2M2NC3	103.00				
1-NC4	103.00				
2-NC4	103.00				
NaOH	40.01				
2-NB	119.10				
NEPD	149.10				
NMP	119.10				
2NC5OH	131.00				
2N2P13PD	161.00				
2N2MB	133.00				
H2SO4	98.00				
Na2SO4	142.00				
2-AB	89.10	5451.65	54.52	5179.07	218.07
AEPD	119.10	404.85		5.23	399.61
AMP	89.10	30.91	20.14	10.46	0.31
2AC5OH	101.00	45.47		5.23	40.24
2A2P13PD	131.00	2.95		0.52	2.43
2A2MB	103.00	46.37		5.23	41.14
RANEY Ni	-----				
H2	2.00				
NH3	17.00				
NiSO4	154.70				
Ca(OH)2	74.10				
CaSO4	176.20				
Ni(OH)2	92.70				
(NH4)2SO4	132.00				
OTHER	-----	393.55	3.94	5.23	384.39
TOTAL lb/batch		6653.77	338.42	5226.68	1088.68
VOLUME(gal)		827.60	41.79	669.52	119.10
TEMP (deg C)		100.00	150.00	150.00	150.00
PRES (mm Hg)		760.00	600.00	300.00	300.00
DENS (lb/gal)		8.04	8.10	7.81	9.14

DATE:2/23/89
2MB & 2AB
CBDAE
WEST HELENA AR

SOLVENT RECOVERY

FROM AMINOALCOHOLS

STREAM NO.		501	502	503
DESCRIPTION		AVERAGE FEED TO COLUMN	RECOVERED CH3OH	COLUMN BOTTOMS
COMPONENTS:	MOL. WT.			
CH2O	30.00			
H2O	18.00	4991.47	39.36	4951.62
CH3OH	32.00	2954.53	2816.80	137.73
1-NC3	89.10			
2-NC3	89.10			
2M2NC3	103.00			
1-NC4	103.00			
2-NC4	103.00			
NaOH	40.01			
2-MB	119.10			
MEPD	149.10			
MMP	119.10			
2MC5OH	131.00			
2M2P13PD	161.00			
2M2MB	133.00			
H2SO4	98.00			
Na2SO4	142.00			
2-AB	89.10	109.58		109.58
AEPD	119.10	4.09		4.09
AMP	89.10	20.45		20.45
2AC5OH	101.00	0.46		0.46
2A2P13PD	131.00	0.03		0.03
2A2MB	103.00	0.47		0.47
RANEY Ni	-----			
H2	2.00			
NH3	17.00			
NiSO4	154.70			
Ca(OH)2	74.10			
CaSO4	176.20			
Ni(OH)2	92.70			
(NH4)2SO4	132.00			
OTHER	-----	10.98		10.98
TOTAL lb/batch		7892.06	2856.65	5235.41
VOLUME(gal)		1021.42	400.94	631.91
TEMP (deg C)		25.00	65.00	65.00
PRES (mm Hg)		760.00	760.00	760.00
DENS (lb/gal)		7.73	6.63	8.28

EXHIBIT D-3

PROCESS DESCRIPTION - NMP

5. NMP (2Nitro-2Methyl-Propanol)

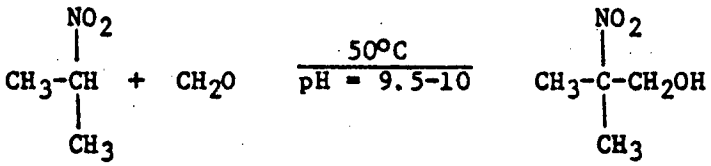
NMP is produced by a condensation reaction between CH_2O and $2\text{-}NC_3$ under carefully controlled conditions of pH and temperature. NaOH is employed to adjust the pH. Following the reaction, the Na^+ ions are removed by ion exchange and the solution is fed to a continuous stripper. The concentrated solution is then fed to the hydrogenator.

The process is illustrated in Figure 5.

The details of the process are as follows:

5.1 Condensation Reaction

a. The chemistry of the reaction is as follows:



b. The operation proceeds as follows: A heel of 44% CH_2O is charged to the reactor. The heel charge is limited to the minimum amount which can be recirculated. The pH is adjusted to 9.5 to 10.0 with 10% NaOH. CH_2O (44%) and $2NC_3$ are simultaneously fed over two hours while the reactor is maintained at a pH of 9.5 to 10.0 and a temperature of 50°C. Recirculation through an external heat exchanger is needed to remove the heat of reaction at a sufficient rate. At the conclusion of the simultaneous feed, the reactor contents are maintained at 50°C and a pH of 9.5-10.0 for four hours.

c. The NMP yield is 97.5%, based on $2NC_3$.

d. The ratio of CH_2O to $2NC_3$ is 1.09. In addition, sufficient CH_2O is fed to completely react the nitroparaffin impurities (i.e. 2 moles CH_2/mole NC_2, 2 mole CH_2O/mole $1\text{-}NC_3$).

e. The NaOH required to maintain the pH is 0.2 wt. % of the batch.

f. The material balance for the condensation reactor is presented in Table 5.1.

5.2 Na^+ Ion Exchange

a. NMP solution from the condensation reactor is passed through an ion exchange column containing strong acid

cation resin; either Rohm & Haas IR-200 (macroreticular) or IR-120 (gel type). The Na^+ level in the solution is reduced to 20 ppm or less and the pH is reduced to 2.5-3.0.

b. The NMP solution fed to the ion exchange column has an average of 1150 ppm Na^+.

c. The process limit of the column effluent is 20 ppm Na^+.

d. The resin is regenerated with a 50% excess of H_2SO_4. The regenerant is fed to the column as a 5% solution.

e. After blowing down the resin with N_2, the liquid hold up on the resin is 50% of the bed volume.

f. At exhaustion, the column is blown down with N_2 and forward washed with 3 bed volumes of H_2O. The forward wash recovers 98% of the NMP solution held up in the resin.

g. After the forward wash, the column is regenerated by the following sequence of operations:

1. N_2 Blowdown
2. H_2O Rinse (2 bed volumes)
3. H_2O Backwash (2 bed volumes)
4. Regeneration (per 5.2 d)
5. H_2O Rinse (8 bed volumes)
6. H_2O Backwash (2 bed volumes)
7. N_2 Blowdown

h. The material balance for Na^+ ion exchange is presented in Table 5.2.

5.3 Nitroalcohol Stripping

a. A continuous stripper is employed to remove excess H_2O (which accompanies CH_2O fed to the reactor) from the NMP solution.

b. The NMP solution is stripped at 70°C under a mild vacuum.

c. The yield across stripping is 99% for NMP.

d. The NMP solution is concentrated to 70%.

e. The material balance for the nitroalcohol stripper is presented in Table 5.4.

5.4 Waste Streams

Wastes from the production of NMP arise from the following:

a. NA^+ Ion Exchange Column Regeneration
(Table 5.2, Stream 113)

b. Nitroalcohol Stripper Overheads
(Table 5.4, Stream 117)

PROCESS DESCRIPTION - AMP

6. AMP (2-Amino-2methyl-propanol)

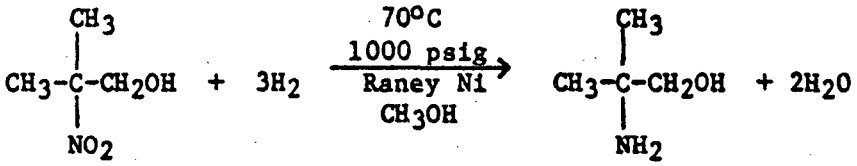
NMP is hydrogenated to AMP in an autoclave over Raney nickel catalyst in a CH_3OH solution. After removing the catalyst by filtration, the AMP solution is fed to an ion exchange column to remove traces of soluble nickel. The AMP solution is stripped and stored. When several batches have been accumulated, the AMP is fed to a batch distillation column for product isolation.

The process is illustrated in Figure 6.

The details of the process are as follows:

6.1 Hydrogenation

a. The chemistry of the reaction is as follows:



b. The autoclave is operated as follows: The 1500 gallon reactor is 40% filled with CH_3OH and a slurry of Raney nickel and is pressurized to 1000 psig with H_2. When the reactor is at the specified pressure and temperature, NMP solution is fed to the autoclave at the rate of 9.2 lbs. NMP/(lb. catalyst hr.). The reaction temperature is maintained at 70°C by recirculating fluid through the internal coils of the autoclave. H_2 is supplied to the autoclave to maintain the reactor at 1000 psig. When the reactor contains 1450 gallons, the NMP feed is stopped. The reactor is vented to a scrubber to 50 psig and the AMP solution is transferred from the autoclave to a catalyst settling tank.

c. The conversion of NMP to AMP is accomplished with a 95% yield.

d. The vent gas from the reactor is chiefly H_2 with traces of CH_3OH, H_2O, NH_3 and amines. The level of amines depends upon the extent to which the unreacted nitroparaffins were stripped from the NMP solution. The vent gas is scrubbed with an H_2SO_4 solution.

e. The concentration of NMP solution fed to the autoclave is about 70%.

f. The material balance for hydrogenation is presented in Table 6.1.

6.2 Catalyst Handling

a. The AMP solution from the catalyst settling tank is passed through a catalyst fines filter which retains the Raney nickel. The solid cakes from the settled catalyst and filtered catalyst are washed with CH_3OH to remove residual AMP solution. The filtered AMP solution and the CH_3OH wash are combined in the Ni^{++} ion exchange column feed tank. The residual catalyst in the settling tank is slurried in CH_3OH and recycled to the Buss reactor catalyst charge tank. In order to maintain the catalyst bed activity, a portion of fresh catalyst equal to the catalyst fines removed is added.

b. The catalyst is pyrophoric, it must be kept moist or isolated from oxygen.

c. The catalyst residue and filter cake are 50% solids.

d. The CH_3OH wash is three times the residue and filter cake volume and recovers 90% of the AMP solution held up on the residue and filter cake.

e. The catalyst slurry from the filter is 25 wt. % solids in CH_3OH.

f. The material balance for catalyst handling is presented in Table 6.2.

6.3 Ni^{++} Ion Exchange

a. The filtered AMP solution contains soluble Ni^{++} which must be removed from the solution. The Ni^{++} is removed by ion exchange with a weak acid resin; Rohm & Haas IRC-50.

b. The AMP solution fed to the column contains an average of 400 ppm Ni^{++}. The solution leaving the column must contain 25 ppm Ni^{++} or less.

c. The resin is regenerated with a 10% excess of H_2SO_4. The regenerant is fed to the column as a 5% solution.

d. The resin swells by 50% upon contact with AMP solution. While this phenomena presents a problem at a laboratory scale, it is expected that the column can operate in a standard downflow manner at a commercial

scale. Alternatively, the resin can be preswelled by feeding denickled AMP in an upflow fashion to the column. The preswelling would occur after regeneration but before the first AMP batch is fed.

e. After blowing down the resin with N_2, the liquid hold up on the resin is 50% of the bed volume.

f. At exhaustion, the column is blown down with N_2 and forward washed with 3 bed volumes of H_2O. The forward wash recovers 98% of the AMP solution held up on the resin.

g. After the forward wash, the column is regenerated by the following sequence of operations:

1. N_2 Blowdown
2. H_2O Rinse (2 bed volumes)
3. H_2O Backwash (2 bed volumes)
4. Regeneration (per 6.3d)
5. H_2O Rinse (8 bed volumes)
6. H_2O Backwash (2 bed volumes)
7. N_2 Blowdown

h. The material balance for Ni^{++} ion exchange is presented in Table 6.3.

6.4 Aminoalcohol Stripping

a. The denickled AMP solution is concentrated in a continuous stripper to a 75% solution.

b. The stripper operates at 100°C and atmospheric pressure.

c. The AMP yield across stripping is 99%.

d. The first batch after Ni^{++} column regeneration is fed directly to the stripper because it is diluted with H_2O from the ion exchange column. The remaining batches before the next regeneration are mixed with dilute AMP solution (from the forward wash of the Ni^{++} column) before being fed to the stripper.

e. The material balance for the aminoalcohol stripper is presented in Table 6.4.

6.5 Distillation

a. Several batches of stripped AMP are accumulated and fed to a batch distillation column. After obtaining a lites cut (to remove residual H_2O), a product cut is

taken overhead to isolate AMP-95. The still bottoms represent a waste stream.

b. The batch distillation column operates at a pot temperature of 150°C.

c. The lites cut is obtained at atmospheric pressure.

d. The product cut is obtained at moderate vacuum; about 600 mm Hg.

e. AMP is recovered at a 95% yield and a purity of 95%.

f. The material balance for batch distillation is presented in Table 6.5.

6.6 Solvent Recovery

a. The aminoalcohol stripper overheads are fed to a batch distillation column to recover CH_3OH for recycle.

b. CH_3OH is recovered at a purity of 98.5% and a yield of 95%.

c. The column operates at 65-70°C and essentially atmospheric pressure.

d. The solvent recovery material balance is presented in Table 6.6.

6.7 Waste Streams

Wastes from the production of AMP arise from the following:

a. Ni^{++} Ion Exchange Column Regeneration (Table 6.3, Stream 223).

b. Solvent Recovery Bottoms (Table 6.6, Stream 503).

c. Distillation Bottoms (Table 6.5, Stream 404).

d. Hydrogenator Vent Scrubber ($(NH_4)_2SO_4$ and amine sulfate sludge).

e. Spent Catalyst (Table 6.2, Stream 214).

f. Spent Catalyst Rinses (H_2O, trace organics).

g. Fresh Catalyst Rinses (H_2O, Al_2O_3).

h. Catalyst Filter Media.

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STREAM NO.		102A BZZL TO REACTOR	102 CH2O TO REACTOR	104 2-MCl TO REACTOR	101 NaOH TO REACTOR	106 DISCHARGE FROM REACTOR
DESCRIPTION						
COMPONENTS:	MOL. WT.					
CH2O	30.00	606.63	4641.16			140.62
H2O	18.00	746.83	5941.42	7.36	497.69	7193.49
CH3OH	32.00	27.66	220.05			247.71
HCl	75.00			88.33		1.10
1-MCl	89.10			125.13		1.56
2-MCl	89.10			14421.00		180.34
2M2NCl	103.00			36.80		36.80
NaOH	40.10				55.32	55.32
NMPD	135.10					155.13
NEPD	149.19					204.16
NMP	119.19					18802.46
H2SO4	98.00					
Na2SO4	142.20					
AMPD	105.10					
AEPD	119.10					
AMP	89.10					
RANEY Ni	-----					
H2	2.00					
NH3	17.00					
NiSO4	154.70					
Ca(OH)2	74.10					
CaSO4	176.20					
Ni(OH)2	92.70					
(NH4)2SO4	132.00					
OTHER	-----			36.80		641.57
TOTAL lb/batch		1383.31	11002.63	14721.43	553.21	27660.28
VOLUME(gal)		148.02	1177.56	1799.13	59.57	2939.91
TEMP (deg C)		35.00	35.00	20.00	20.00	50.00
PRES (mm Hg)		760.00	760.00	760.00	760.00	760.00
DENS (lb/gal)		9.34	9.34	8.18	9.29	9.22

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ION EXCHANGE
30 CU FT RESIN: 3 BATCH /CYCLE

STREAM NO.		110A	110	111	112	113	114	116	115	117	118	119
DESCRIPTION		B/N 1 FROM I.E. COLUMN	B/N 2-3 FROM I.E. COLUMN	FORWARD WASH TO COLUMN	FORWARD WASH FROM COLUMN	RINSE #1 TO COLUMN	BACKWASH #1 TO COLUMN	H2SO4 TO COLUMN	H2O TO DILUTE ACID	RINSE #2 TO COLUMN	BACKWASH #2 TO COLUMN	TOTAL WASTE STREAM
COMPONENTS:	MOL. WT.											
CH2O	30.00	135.66	140.62		4.96							[illegible]
HCO	19.00	7685.12	7218.33	5615.24	4965.47	3743.49	3743.49	22.74	5716.86	14573.97	3743.49	51227.04
CH3CH	32.00	238.45	247.71		8.78							8.43
NC2	75.00	1.06	1.10		0.04							0.04
1-NC3	89.10	1.61	1.66		0.06							0.06
2-NC3	89.10	171.59	180.34		6.35							0.36
2M2NC3	103.00	35.43	36.80		1.30							0.07
NaOH	40.10											
NMPD	135.10	149.33	155.13		5.50							0.31
NEPD	149.10	196.52	204.16		7.23							0.40
NMP	119.10	18099.13	18802.46		666.29							37.04
H2SO4	98.00							302.09				99.29
Na2SO4	142.20											294.26
AMPD	105.10											
AEPD	119.10											
AMP	89.10											
RANEY Ni	-----											
H2	2.00											
NH3	17.00											
NiSO4	154.70											
Ca(OH)2	74.10											
CaSO4	176.20											
Ni(OH)2	92.70											
(NH4)2SO4	132.00											
OTHER	-----	617.57	641.57		22.73							1.26
TOTAL lb/batch		27533.07	27629.79	5615.24	5688.77	3743.49	3743.49	324.82	5716.86	14573.97	3743.49	32360.80
VOLUME(gal)		3001.51	3002.45	673.29	670.18	448.86	448.86	21.93	685.48	1795.44	448.86	3810.94
TEMP (deg C)		45.00	45.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00
PRES (mm Hg)		760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00
DENS (lb/gal)		9.17	9.20	8.34	8.49	8.34	8.34	14.81	8.34	8.34	8.34	8.49

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NITRO ALCOHOL STRIPPER

STREAM NO.		110	110	122	123	110	120	155	122	123
		AVERAGE	S/N 1	NA	NA	S/N 2-3	DILUTE	FEED	NA	NA
DESCRIPTION		NMP FROM COLUMN	NMP FROM COLUMN	STRIPPER OVERHEAD	STRIPPER BOTTOM	NMP FROM COLUMN	NA TO STRIPPER	TO NA STRIPPER	STRIPPER OVERHEAD	STRIPPER BOTTOMS
COMPONENTS:	MOL. WT.									
CH2O	30.00	136.87	135.36	16.92	118.44	140.62	3.49	143.10	17.89	125.21
H2O	18.00	7440.59	7855.12	1577.02	6308.10	7218.33	2492.74	9701.06	2810.31	6890.75
CH3OH	32.00	244.52	238.45	238.21	0.24	247.71	4.38	252.10	251.85	0.25
NC2	75.00	1.09	1.08	1.08	0.01	1.13	0.01	1.12	1.11	0.01
1-NC3	89.10	1.54	1.51	1.49	0.02	1.56	0.03	1.59	1.58	0.02
2-NC3	89.10	176.09	173.59	171.86	1.74	180.34	3.20	183.53	181.70	1.84
2M2NC3	103.00	36.34	35.43	35.07	0.35	36.80	0.65	37.46	37.09	0.37
NaOH	40.10									
NMPD	135.10	153.20	149.33	0.75	148.58	155.13	2.75	157.88	0.79	157.09
HEPD	149.10	201.62	196.52	0.98	195.54	204.16	3.62	207.78	1.04	206.74
NMP	119.10	18568.02	18099.13	180.99	17918.14	18802.46	333.14	19135.60	191.36	18944.25
H2SO4	98.00									
Na2SO4	142.20									
AMPD	105.10									
AEPD	119.10									
AMP	89.10									
RANEY Ni	-----									
H2	2.00									
NH3	17.00									
NiSO4	154.70									
Ca(OH)2	74.10									
CaSO4	176.20									
Ni(OH)2	92.70									
(NH4)2SO4	132.00									
OTHER	-----	633.57	617.57		617.57	641.57	11.37	652.93		652.93
TOTAL lb/batch		27597.55	27533.07	2224.34	25308.73	27629.79	2844.39	30474.18	3497.70	25976.48
VOLUME(gal)		3000.14	3001.51	269.75	2930.80	3002.45	335.09	3335.90	422.59	2933.04
TEMP (deg C)		35.00	35.00	70.00	70.00	35.00	25.00	35.00	70.00	70.00
PRES (mm Hg)		760.00	760.00	225.00	225.00	760.00	760.00	760.00	225.00	225.00
DENS (lb/gal)		9.20	9.17	8.25	9.20	9.20	8.48	9.14	8.28	9.20

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				HYDROGENATOR						
						H2 BATCH	PER COND	BATCH =	3.26	
STREAM NO.				201	202	203	204	205	206	207
DESCRIPTION		AVG NMP TO HYDROG		CH3OH TO REAC	CATALYST TO REAC	CATALYST LINE RINSE	NMP TO REAC	HYDROGEN TO REAC	VENT FROM REAC	DISCHAR FROM REAC
COMPONENTS:	MOL. WT.									
CH2O	30.00	122.36					37.46			
H2O	18.00	6894.58		6.73	79.62	0.99	2039.69			3888.3
CH3OH	32.00	0.25		442.09	1378.28	65.01	0.08			1885.4
NC1	75.00	0.01					0.00			
1-NC3	89.10	0.02					0.00			
2-NC3	89.10	1.80					0.55			
DM2NC3	103.00	0.37					0.11			
NaOH	40.10									
NMPD	135.10	154.25					47.00			
NBPD	149.10	203.01					61.85			
NMP	119.10	18602.21					5667.73			
H2SO4	98.00									
Na2SO4	142.20									
AMPD	105.10				0.13					34.7
ABPD	119.10				0.17					46.9
AMP	89.10				14.69					4028.0
RANEY Ni	-----				436.30					436.3
H2	2.00							294.25	4.10	
NH3	17.00								0.41	
NiSO4	154.70									
Ca(OH)2	74.10									
CaSO4	176.20									
Ni(OH)2	92.70									
(NH4)2SO4	132.00									
OTHER	-----	641.14			1.70		195.34		0.67	465.1
TOTAL lb/batch		26420.56		448.82	1909.89	66.00	8049.82	294.25	5.18	10763.60
VOLUME(gal)		2871.86		68.00	222.00	10.00	875.00			1315.42
TEMP (deg C)		70.00		25.00	25.00	25.00	25.00			45.00
PRES (mm Hg)		760.00		760.00	760.00	760.00	760.00			
DENS (lb/gal)		9.20		6.60	8.60	6.60	9.20			8.15

DATE:02/23/89
NMP & AMP
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WEST HELENA A2

CATALYST HANDLING SYSTEM
H2 BATCH per cond BATCH = 3.29

STREAM NO.		208	209	210	211	212	213	214	215
DESCRIPTION		AMP FROM DECANT TANK	CH3OH WASH TO TANK	WASH FROM TANK	TOTAL AMP FROM TANK	CH3OH FOR SLURRY	SLURRY FROM TANK	SPENT CATALYST REMOVED	FRESH CATALYST ADDED
COMPONENTS:	MOL. WT.								
CH2O	30.00								
H2O	18.00	3710.18	5.77	146.57	3856.75	13.32	38.83	3.89	42.72
CH3OH	32.00	1899.03	573.92	429.82	2236.85	1308.99	1531.41	153.14	
NC2	75.00								
1-NC3	89.10								
2-NC3	89.10								
2M2NC3	103.00								
NaOH	40.10								
NMPD	135.10								
NEPD	149.10								
NMP	119.10								
H2SO4	98.00								
Na2SO4	142.20								
AMPD	105.10	33.33		1.27	34.59		0.14	0.01	
ABPD	119.10	45.03		1.71	46.75		0.19	0.02	
AMP	89.10	3664.80		146.95	4011.75		16.33	1.63	
RANEY Ni	-----						436.30	43.63	43.63
H2	2.00								
NH3	17.00								
NiSO4	154.70								
Ca(OH)2	74.10								
CaSO4	176.20								
Ni(OH)2	92.70								
(NH4)2SO4	132.00								
OTHER	-----	446.32		16.97	463.29		1.89	0.19	
TOTAL lb/batch		9908.69	584.69	743.29	10651.98	1328.83	2025.15	202.51	87.26
VOLUME(gal)		1255.12	98.59	102.25	1356.77	201.34	239.48	23.95	7.46
TEMP (deg C)		40.00	25.00	25.00	40.00	25.00	25.00	25.00	25.00
PRES (mm Hg)		760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00
DENS (lb/gal)		7.89	6.60	7.27	7.85	6.60	8.46	8.46	11.67

E:02/23/89
& AMP
AE
T HELENA AB

---------- Ni ION EXCHANGE ----------
50 CU FT RESIN: 8 BATCH /CYCLE

3.28

EAM NO.		x 211 AMP	220A S/N 1	220 S/N 2-8	221 FORWARD	222 FORWARD	223 RINSE #1	224 BACKWASH	226 H2SO4	225 H2O TO	227 RINSE #2	226 BACKWASH	229 TOTAL
SCRIPTION		FED TO	FROM I.E.	FROM I.E.	WASH TO	WASH FROM	TO	#1 TO	TO	DILUTE	TO	#2 TO	WASTE
		COLUMN	COLUMN	COLUMN	COLUMN	COLUMN	COLUMN	COLUMN	COLUMN	ACID	COLUMN	COLUMN	STREAM
PONENTS:	MOL. WT.												
O	30.00												
	18.00	12658.35	13431.40	12669.07	14038.10	12479.92	9353.73	9358.73	37.78	6987.29	24956.62	6239.15	58175.88
OH	32.00	7346.19	6855.27	7346.19		453.66							3.25
	75.00												
CO	89.10												
C3	89.10												
NCO	103.00												
H	40.10												
O	135.10												
O	149.10												
	119.10												
O4	98.00								369.22				135.74
SO4	142.20												
O	105.10	113.54	106.39	113.54		7.01							0.14
O	119.10	153.43	143.76	153.43		9.47							0.19
	89.10	13167.10	12337.60	13167.10		812.91							16.59
ET Ni	-----												
	2.00												
	17.00												
O4	154.70												368.55
CHI2	74.10												
O4	176.20												
CHI2	92.70												
4)2SO4	132.00												
ER	-----	1520.56	1407.29	1503.08		93.68							19.40
AL lb/batch		34961.17	34311.71	34954.41	14038.10	13856.86	9358.73	9358.73	397.01	6987.29	24956.62	6239.15	58725.43
UME(gal)		4453.10	4358.56	4452.76	1683.23	1676.81	1122.15	1122.15	26.80	837.81	2992.40	748.10	6911.69
P (deg C)		40.00	30.00	35.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00
E (mm Hg)		760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00
E (lb/gal)		7.85	7.87	7.85	8.34	8.26	8.34	8.34	14.81	8.34	8.34	8.34	8.50

DATE:02/23/89
NMP & AMP
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		AMINOALCOHOL STRIPPER							
STREAM NO.		220A	232A	233A	220	230	231	232	233
		B/N 1	B/N 1 AA	B/N 1 AA	B/N 2-8	DIL. AMP	FEED	AA	AA
DESCRIPTION		TO AA	STRIPPER	STRIPPER	TO FEED	TO FEED	TO AA	STRIPPER	STRIPPER
		STRIPPER	OVERHEAD	BOTTOMS	TANK	TANK	STRIPPER	OVERHEAD	BOTTOMS
COMPONENTS:	MOL. WT.								
CH2O	30.00								
H2O	18.00	13431.49	12759.83	671.67	12669.37	1782.85	14451.21	13780.18	671.03
CH3OH	32.00	6883.27	6816.42	66.85	7348.13	64.81	7413.00	7346.87	66.13
NC3	75.00								
1-NC3	89.10								
2-NC3	89.10								
2M2NC3	103.00								
NaOH	40.10								
NMPD	135.10								
NEPD	149.10								
NMP	119.10								
H2SO4	98.00								
Na2SO4	142.20								
AMPD	105.10	196.39	1.06	105.32	113.54	1.00	114.54	1.15	113.39
AEPD	119.10	143.76	1.44	142.32	153.43	1.35	154.78	1.55	153.23
AMP	89.10	12337.60	123.38	12214.22	13167.10	116.13	13283.23	132.83	13150.40
RANEY Ni	-----								
H2	2.00								
NH3	17.00								
NiSO4	154.70								
Ca(OH)2	74.10								
CaSO4	176.20								
Ni(OH)2	92.70								
(NH4)2SO4	132.00								
OTHER	-----	1407.29	14.07	1393.22	1503.08	13.41	1515.49	15.16	1501.33
TOTAL lb/batch		34311.71	19716.20	14595.51	34954.41	1979.55	36933.96	21218.88	15715.08
VOLUME(gal)		4358.56	2548.51	1811.79	4452.76	239.54	4691.68	2742.83	1950.73
TEMP (deg C)		25.00	100.00	100.00	25.00	25.00	25.00	100.00	100.00
PRES (mm Hg)		760.00	760.00	760.00	760.00	760.00	760.00	760.00	760.00
DENS (lb/gal)		7.87	7.74	8.06	7.85	8.26	7.87	7.74	8.06

DATE:02/23/88
NMP & AMP
CEDAR
WEST HELENA AR

PRODUCT DISTILLATION

STREAM NO.		401	402	404	405
DESCRIPTION		FEED TO DIST	LITES OUT	PRODUCT OUT	DIST BOTTOMS
COMPONENTS:	MOL. WT.				
CH2O	30.00				
H2O	18.00	715.31	109.03	598.77	7.15
CH3OH	32.00	73.36	73.36		
NC3	75.00				
1-NC3	89.13				
2-NC3	89.10				
2M2NC3	103.00				
NaOH	40.10				
NMPD	135.19				
NEPD	149.19				
NMP	113.19				
H2SO4	98.00				
Na2SO4	142.20				
AMPD	105.19	112.24		13.02	99.22
AEPD	119.19	151.68		13.02	138.66
AMP	89.19	13016.66	130.17	12365.83	520.67
RANEY Ni	-----				
H2	2.00				
NH3	17.00				
NiSO4	154.70				
Ca(OH)2	74.10				
CaSO4	176.20				
Ni(OH)2	92.70				
(NH4)2SO4	132.00				
OTHER	-----	1485.88	14.86	26.03	1444.99
TOTAL lb/batch		15555.14	327.79	13016.66	2210.69
VOLUME(gal)		1930.89	41.96	1661.89	234.97
TEMP (deg C)		25.00	150.00	150.00	150.00
PRES (mm Hg)		760.00	760.00	600.00	600.00
DENS (lb/gal)		8.06	7.81	7.83	9.41

DATE:[illegible]
HMP & AMP
CEDAR
WEST HELENA AR

STREAM NO.		501	502	503
DESCRIPTION		AVERAGE FEED TO COLUMN	RECOVERED OVHDS	COLUMN BOTTOMS
COMPONENTS:	MOL. WT.			
TMCO	[illegible]			
HCl	18.00	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
HOC	78.00			
1-HCO	99.11			
2-HCO	99.11			
CH3HCl	100.00			
NaOH	40.00			
NMEI	105.11			
MEPD	143.19			
HMP	119.11			
H2SO4	98.09			
Na2SO4	142.03			
AMPD	105.19	1.13		1.13
AEPD	119.19	1.51		1.51
AMP	89.11	261.65		261.65
RANEY Ni	-----			
H2	2.00			
NH3	17.03			
NiSO4	154.70			
Ca(OH)2	74.10			
CaSO4	176.20			
Ni(OH)2	92.70			
(NH4)2SO4	132.00			
OTHER	-----	29.87		29.87
TOTAL lb/batch		21332.00	7339.29	13992.71
VOLUME(gal)		2757.03	1107.57	1651.83
TEMP (deg C)		25.00	65.00	65.00
PRES (mm Hg)		760.00	760.00	760.00
DENS (lb/gal)		7.74	6.63	8.32

EXHIBIT E-1

1. Tris (Hydroxy Methyl) Amino Methane

a. Nitromethane

Assay	97% min. by weight
Nitroethane Content	2% max. " "
1-Nitropropane Content	0.1% max. " "
2-Nitropropane Content	0.1 max. " "
Acidity	0.1% max. " "
Water	0.1% max. " "
Others	1.0% max. " "

b. Methanol — 99.85% (minimum)

c. Formaldehyde — 44.00% (minimum)

d. Raney Nickel — 3100 grade (Davison Chemical)

e. Sodium Hydroxide — 49-51%

f. Sulfuric Acid — 92.5-94.0%

g. Hydrogen — 99.7% (minimum)

h. Decolorizing Carbon — Calgon APA 12x40 granular

i. Weak Exchange Resin — R&H FRC-50

j. Strong Cation Exchange Resin — R&D IR-200

RCZ:doc
2/13/89
3387K

EXHIBIT E-2

2. Racemic 2-Amino-1-Butanol

a. 1-Nitropropane

Assay	94% min. by weight
Nitromethane Content	0.1% max. " "
Nitroethane Content	1.0% max. " "
2-Nitropropane Content	5.0% max. " "
Acidity (as acetic acid)	0.1% max. " "
Water	0.1% max. " "
Others	1.0% max. " "

b. Methanol — 99.85%

c. Formaldehyde — 44.00%

d. Raney Nickel — 3100 grade (Davison Chemical)

e. Sodium Hydroxide — 49-51%

f. Sulfuric Acid — 92.5-94.0%

g. Hydrogen — 99.7% (minimum)

h. Weak Exchange Resin — R&H FRC-50

i. Strong Cation Exchange Resin — R&D IR-200

RCZ:doc
2/13/89
3387K

EXHIBIT E-3

3. 2-Amino-2-Methyl-1-Propanol

a. 2-Nitropropane

Assay	94% min. by weight
Nitromethane Content	0.1% max. " "
Nitroethane Content	3.0% max. " "
1-Nitropropane Content	5.0% max. " "
Acidity	0.1% max. " "
Water	0.1% max. " "
Others	1.0% max. " "

b. Methanol — 99.85% (minimum)

c. Formaldehyde — 44.00% (minimum)

d. Raney Nickel — 3100 grade (Davison Chemical)

e. Sodium Hydroxide — 49-51%

f. Sulfuric Acid — 92.5-94.0%

g. Hydrogen — 99.7% (minimum)

h. Weak Exchange Resin — R&H FRC-50

i. Strong Cation Exchange Resin — R&D IR-200

RCZ:doc
2/13/89
3387K

EXHIBIT F

Raw Material Assay Procedures

- GC Analysis of 1-Nitropropane and 2-Nitropropane

- GC Analysis of Nitromethane and Nitroethane

- Determination of Acidity in Nitroparaffins

- Formaldehyde Assay

- Sulfuric Acid Assay

- Sodium Hydroxide Assay

REAGENTS

Nitromethane	99+%	Aldrich (Gold Label)
Nitroethane	99.5+%	Aldrich (Gold Label)
1-Nitropropane	98%	Aldrich
2-Nitropropane	97%	
2-Methyl-2-Nitropropane		Aldrich
1-Nitrobutane		Aldrich
Butanol	99.9%	Fisher, HPLC grade
Methanol	99.9%	Aldrich (Gold Label)

Standards should be assayed prior to use.

STANDARD PREPARATION

Prepare a primary standard from assayed quantities from assayed. Quantities of the listed Nitroparaffins weigh into a 100 ml vial 1% by weight, Nitromethane, Nitroethane, 1-Nitropropane, 2-Nitropropane. Suggested weights 1.0000 gm. Add 1.0000 gm of Butanol to vial. Add methanol until total weights equals 100 gms. Mix well. Calculate the wt/wt% of each component.

SAMPLE PREPARATION

The nitropropanes must be diluted prior to the GC assay. This is to better define the peak shapes.

1-Nitropropane and 2-Nitropropane are diluted in a 1:10 dilution. To do this, weigh exactly 1.0gm of the NP sample into a 10ml vial. Weight in 0.1000gm of Butanol. Dilute the sample by adding methanol until total weight equals 10gm.

PROCEDURE

Inject 0.5 ul of the standard and repeat until two chromatograms are duplicated. Inject 0.5 ul of the sample. If desired, the standard or sample may be injected again after the sample analysis (to assure reproducibility).

8. CALCULATIONS

Calculate new response factors for each sample analysis. A response factor need not be claculated for the primary peak of interest. Equations needed are:

(a) $\frac{\text{Component area in Standard}}{\text{Internal Standard area in Standard}}$ = Standard Area Ratio

(b) $\frac{\text{Average of Standard Area Ratio}}{\text{wt/wt\% Component Standard}}$ = Component Response Factor

(c) $\frac{\text{Component Area in Sample}}{\text{Internal Standard Area in Sample}}$ = Sample Area Ratio

For 1-Nitropropane Samples:

(d) $\frac{\text{Average of Sample Area Ratio}}{\text{Component Response Factor}} \cdot 2$ = wt/wt% Component in Sample

For 2-Nitropropane Samples:

(d) $\frac{\text{Average of Sample Area Ratio}}{\text{Component Response Factor}} \cdot 10$ = wt/wt% Component in Sample

(e) Assay of Product = 100% - Sum of all wt/wt% Component Impurities in Sample - H_2O determined by Karl Fisher
Nitroparaffin

Report the assay of the nitropraffin and major impurities in the sample. Record response factors used in the instrument log along with the Sample Number.

9. TEST STATISTICAL VALIDATION

(a) 2 sigma deviation -
(b) lower limit of detection -
(c) Total test time -

10. REFERENCES

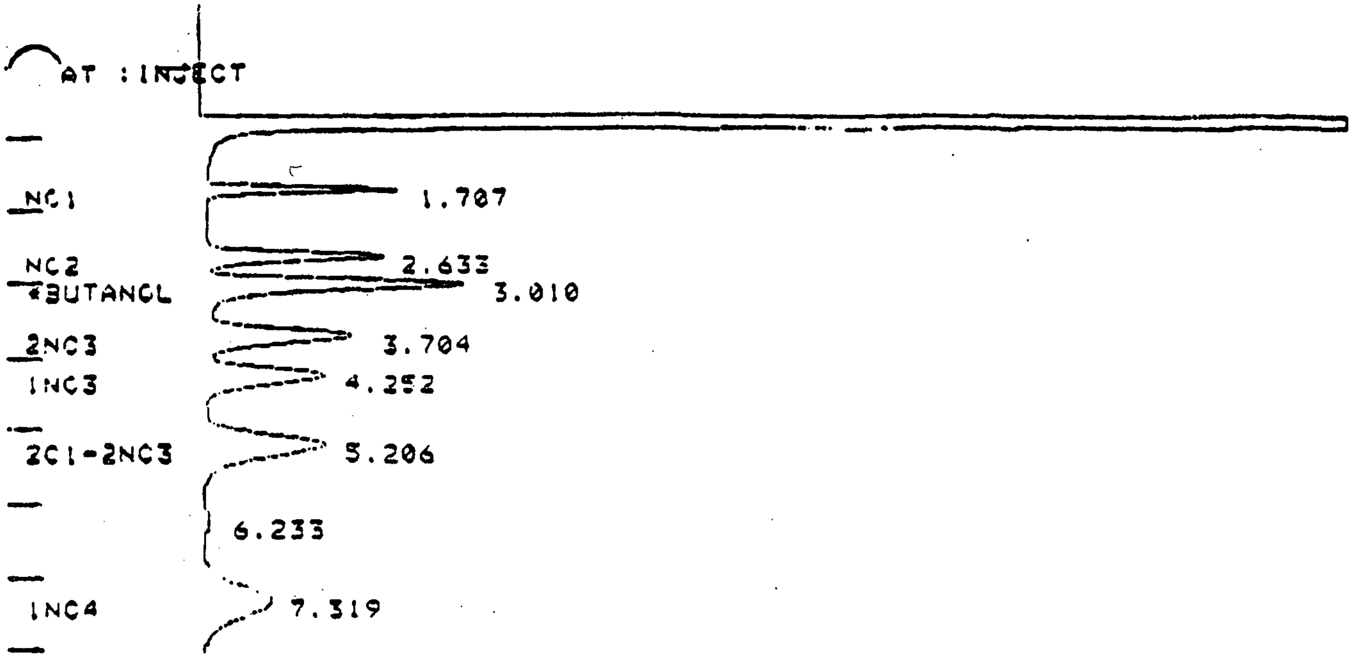
(a) Laboratory work by C. S. Smith, in Nashua, OCD, August, 1983.

Written by : Cheryl J Smith

Reissued by: Marsha Hurtl Gld

.32

CHART SPEED 1.0 CM/MIN
ATTEN: 64 ZERO: 5% 1 MIN/TICK



TITLE: GC ANALYSIS OF 1NC3 AND 2NC3 14:05 13 MAR 86

CHANNEL NO: 1 SAMPLE: STD METHOD: NPAP-27

PEAK NO	PEAK NAME	RESULT FACTOR	TIME (MIN)	TIME OFFSET	AREA COUNTS	SEP CODE	W1/2 (SEC)
1	NC1	2.864130U	1.707	-0.013	53138	BB	5.15
2	NC2	1.783220U	2.633	-0.017	81460	BV	8.35
3	*BUTANOL	INT STDU	3.010	-0.020	148683	VV	10.00
4	2NC3	1.410860U	3.704	-0.036	104330	VV	12.95
5	1NC3	1.484580U	4.252	-0.038	97191	VV	14.80
6	2C1-2NC3	1.175810U	5.206	-0.044	127795	VV	19.30
7			6.233		950	T	20.00
8	1NC4	1.434760U	7.319	-0.091	101920	VV	27.65
TOTALS:				-0.259	715377		

DETECTED PKS: 9 REJECTED PKS: 0

AMT STD: 1.10950 MULTIPLIER: 1.00000

NOISE: 0.0 OFFSET: 235

SAVED FILE: NPAP27103

NOTES:
COLUMN 8'X1/8" 316 O.D. SS WITH 100/120 MESH POROPAK Q OR QS PACKING. HELIUM AT 30 ML/MIN. FLOW RATE. INJECTION SIZE IS 1/2 MICROLITERS. REPORT WT/WT% ASSAY OF NITROPARAFFINS AND MAJOR IMPURITIES IN THE SAMPLE FOR 1NC3 AND 2NC3 PURITY.

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SINGLE CHANNEL METHOD: NPAP-27 14:14 13 MAR 86

SECTION 1: BASIC
PAGE 1
ANALYSIS PARAMETERS
CHANNEL: 1
CALCULATION: IS
AREA/HT: A
STOP TIME: 15.00
NUMB EXPECTED PKS: 40
EQUILIBRATION TIME: 0
UNRETAINED PK TIME: 0.00
UNIDENT PK FACTOR: 0.000000
SLICE WIDTH: 10

PAGE 2
SAMPLE PARAMETERS
RUN TYPE: C
SAMPLE ID: STD
DIVISOR: 1.000000
AMT STD: 1.109500
MLTPLR: 1.000000

PAGE 3
REPORT INSTRUCTIONS
WHERE TO REPORT: L
COPIES: 1
TITLE: GC ANALYSIS OF 1NC3 AND 2NC3
FORMAT: E
DECIMAL PLACE: 4
RESULT UNITS: %%%%%%
REPORT UNIDENT PKS: Y
REPORT INSTRUMENT CONDITIONS: N

PAGE 4
PLOT INSTRUCTIONS
PLOT: Y
ZERO OFFSET: 5
ANNOTATION
RETENTION TIME: Y
PLOT CONTROL: Y
TIME TICKS: Y
TIME EVENTS: N
PK START/END: N

PAGE 5
CHART SPEED
PAGES OR CM/MIN: C
INIT VALUE: 1.0

PAGE 6
PLOT ATTEN
INIT PLOT ATTEN: 64

SECTION 2: TIME EVENTS
PAGE 1

LINE#	TIME	EVENT	VALUE
1	0.00	PR	100
2	0.00	SN	2
3	0.00	T%	5.0
4	0.00	WI	9
5	0.46	II	1.10
6	4.57	WI	18
7	10.58	WI	36

SECTION 3: PEAK TABLE
PAGE 1
STD PK#: 4
RELATIVE RETEN PK#: 0
RESOLUTION PK#: 0

%: 10
MIN: 0.00
NON REF
%: 0
MIN: 0.00

PAGE 2

PK#	TIME	NAME	FACTOR	AMOUNT	REF	GR#	MUST LO	MUST HI
1	0.98	MEOH	0.000000	1.000000			0.000000	0.000000
2	1.71	*NC1	2.864130	1.135720			0.000000	0.000000
3	2.63	*NC2	1.783280	1.084000			0.000000	0.000000
4	3.01	**BUTANOL	1.000000	1.109500			0.000000	0.000000
5	3.70	*2NC3	1.410860	1.098400			0.000000	0.000000
6	4.25	*1NC3	1.484580	1.076700			0.000000	0.000000
7	5.21	*2C1-2NC3	1.175810	1.120500			0.000000	0.000000
8	7.32	*1NC4	1.434760	1.091200			0.000000	0.000000

SECTION 4: GC INSTRUMENT CONTROL
PAGE 1
COL TEMP
ISO/INIT COL TEMP: 240
INIT HOLD TIME: 20.00

PAGE 2
DETECTORS
DET A TYPE: FID
DET B TYPE:

LN#	TIME	SIDE	ATTN	RANGE	ZERO
1	0.00	A	32	10	Y
2	0.00	B			Y

PAGE 3
TEMP/FLOW
INJ A TEMP: 240
INJ B TEMP: 130
ION TEMP: 0
TCD TEMP: 0
TCD FIL TEMP: 0
AUX TEMP: 0
COL A FLOW: 0
COL B FLOW: 0

PAGE 4

SECTION 7: POST RUN
PAGE 1
FILE NAME: NPAP27
SAVE INSTRUCTIONS
TYPE: RAW
WHERE TO SAVE: L
TRANSMIT REPLOT INSTRUCTIONS
TRANSMIT RAW DATA: N
REPLOT WITH BASELINES: N
RAW DATA LOCATION: L
TRANSMIT REPORT: N

PAGE 2
METHOD LINKING INSTRUCTIONS
METHOD:
LINK CALC RESULTS: N
PROGRAM EXECUTION
PROGRAM:
PARAMETERS:
RESERVE PRINTER: Y

SECTION 10: NOTE PAD
PAGE 1

LINE#	VALUE
1	COLUMN 3'X1/8" 316 O.D. SS WITH 100/120
2	MESH POROPAK Q OR QS PACKING. HELIUM AT
3	30 ML/MIN. FLOW RATE. INJECTION SIZE IS
4	1/2 MICROLITERS. REPORT WT/WT% ASSAY OF
5	NITROPARAFFINS AND MAJOR IMPURITIES IN

NITROPARAFFINS

ANALYTICAL PROCEDURE

NUMBER: NPAP-12

TITLE: Gas Chromatographic Assay of Nitromethane and Nitroethane

ISSUE NO.: 4

REASON FOR REISSUE: Updating Method

I. SCOPE

This method serves as a guide to the assay of Nitromethane and Nitroethane using packed column gas chromatography. HAZARDOUS MATERIALS ARE INVOLVED. SPECIFIC PRECAUTIONARY STATEMENTS ARE GIVEN IN APPENDIX 1.

II. APPLICABLE DOCUMENTS

Material Safety Data Sheets (Appendix 1)

ASTM Standards:

E260 Standard Practice for Packed Column Gas Chromatography

E355 Recommended Practice for Gas Chromatography Terms & Relationships

E594 Recommended Practice for Testing Flame Ionization Detectors Used in Gas Chromatography

(All applicable ASTM standards are available in the laboratory.)

III. TERMINOLOGY

A representation of the apparatus is labeled in Appendix 2.

Carrier Gas: Helium

Flow Controller: The Flow Controller maintains the flow rate of 30 mls/min.

Injection Port: The sample is introduced to the system through a septum and into the injection port.

Column: The column is 8' x 1/8" O.D. 316 SS with 100/120 mesh Porapak.

Oven: The oven maintains the specified column temperature. An initial temperature of 150°C is maintained for 1 minute. Then the temperature is increased 12°C per minute until a final temperature of 240°C is attained. This temperature is held for 10 minutes.

Detector: A flame ionization detector is used.

Interface: The technician addresses the apparatus through the interface.

Integrator Data System Computer: The Varian Gas Chromatography Apparatus provides a differential record of the sample as well as numerical data.

IV. SUMMARY OF PRACTICE

A. Preparation of Standard

1. A 50 ml vial is placed on the balance. Approximately .5g of each of the following is added to the vial: Acetaldehyde, Acetonitrile, Propionitrile, Nitromethane, Nitroethane, 1-Nitropropane, 2-Nitropropane, 2-methyl-2-Nitropropane, 1-Nitrobutane, Ethanol, and N-butanol.

2. Enough methanol is added to give a volume of approximately 50 ml. The weight of the added methanol is recorded.

3. Each of the components of the standard is analyzed for impurities. The analyses are used to correct the weight of each component of the standard. For example, assume the Nitromethane used in the standard is found to contain 3% Propionitrile. Also assume the weight of Nitromethane added is .5500g and the weight of Propionitrile added is .4700g. To find the corrected weight:

   .97 (.5500g) = .5335g Nitromethane
   .03 (.5500g) + .4700g = 4865g Propionitrile

   The calculations are applied to attribute all impurities to the proper component of the standard.

4. The corrected weights are then added.

5. Then each corrected weight is divided by the weight found in Step #4. This value is multiplied by 100.

6. The result is an "amount" of each component.

7. The "amounts" are entered in Section 3, page 2 of Method Modify (Appendix 3).

8. All factors except butanol are reset to zero. Butanol is set at 1.0 and serves as an internal standard.

9. The prepared standard solution is then injected in Calibration Mode.

10. The system is then ready for use. The standard should be reinjected daily. More information on operating parameters is given in Appendix 4.

B. Preparation of Sample

The sample is prepared using a Brinkmann Diluter. The solution in the diluter bottle is 1000 ml Methanol and 10g of Butanol.

After dilution, 1 microliter of sample is injected.

V. CALCULATIONS

Results of the assay are given in percentage of total weight contributed by each component detected. The sum of all percentages is also given. When this sum does not equal 100, the valves may be corrected by the following method:

1. Divide 100 by the given total percentage.

2. Multiply each percentage given by the factor obtained in Step #1.

The sum of the corrected percentages will be 100.

Note: When the given sum of the percentages is more than $\pm$ 3% from 100%, the method may need to be modified by reinjection of the standard or preparation of a new standard.

VI. REFERENCES

ASTM Standards E260, E355, E594
Grace Material Safety Data
Lab Technician Work at Nitroparaffins Division, Deer Park, TX

Written By:_____

Approved By: L L Mayo

0212m

NITROPARAFFINS

ANALYTICAL PROCEDURE

NUMBER: NPAP-11

TITLE: Determination of Acidity as Acetic Acid (CH_3COOH)

ISSUE NO.: 2

REASON FOR REISSUE: Update of Method

I. SCOPE

This method covers the determination of total acidity as acetic acid in organic compounds and hydrocarbon mixtures. It is applicable to the nitroparaffins. The method determines acidity at concentrations below .05%. HAZARDOUS MATERIALS ARE INVOLVED. SPECIFIC PRECAUTIONARY STATEMENTS ARE GIVEN IN APPENDIX 1.

II. APPLICABLE DOCUMENTS

Material Safety Data Sheets (Appendix 1)
ASTM Standards:

D1613 Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer and Related Products.

E200 Preparation, Standardization, and Storage of Standard Solutions in Chemical Analysis.

III. SUMMARY OF METHOD

The sample is mixed with an equal volume of alcohol and titrated with sodium hydroxide solution to the blue bromocresol green end point.

IV. SIGNIFICANCE AND USE

This method is useful for determining low levels of acidity in hydrocarbon mixtures. Monitoring acidity of products aids in maximizing plant potential.

V. REAGENTS

Alcohol - Methanol. CAUTION: EXPOSURE TO METHANOL MAY CAUSE HEADACHE, NAUSEA, AND BLINDNESS .

Bromocresol Green - One gram of bromocresol green in 20 ml isopropanol. Dilute to 100 ml with isopropanol.

Sodium Hydroxide - standard solution .01N: Dissolve .4 grams NaOH in methanol and dilute to 1 liter. Ethanol also may be used.

VI. PROCEDURE

1. Measure 25 ml alcohol into 125 ml Erlenmeyer flask.
2. Add 5 drops bromocresol green indicator. Solution should turn bright yellow.
3. Add enough titrant, 0.01N NaOH in methanol, to reach bromocresol green endpoint (deep blue).
4. Add approximately 25 ± 0.01g of sample.
5. Add titrant to reach bromocresol green endpoint (deep blue).
6. Record volume of titrant used.

VII. CALCULATIONS

A. Calculation of weight %, acetic acid:

$$\frac{V(N)\ (.06)\ (100)}{W} = \text{Weight \% Acidity, as acetic acid}$$

V = Volume of titrant

N = Normality of titrant

W = Weight of sample added

B. Calculation of Normality of NaOH solution:

Weigh 0.01 ± 0.0001g of potassium biphthalate which has been dried at 120°C for two hours in a 250 ml Erlenmeyer flask. Dissolve in 50 ml of water, add 3 drops phenolphthalein indicator and titrate to a permanent pink end point. Repeat with two more samples of potassium biphthalate.

$$\text{Normality of} = \frac{(\text{g Potassium Biphthalate})\ (4.8792)}{(\text{ml NaOH titrated})}$$

Written By: Scott DeLaney

Approved By: _____

0216m

 Note: Assay 0078B can be used in place of this procedure with appropriate modifications. This procedure can not be used in place of 0078B. SAP 0078C

ORGANIC CHEMICALS DIVISION

HAMPSHIRE CHEMICAL

STANDARD ANALYTICAL PROCEDURE

Number: 0078C Date: September 15, 1976

Formaldehyde Assay

Principle of Method:

Liberation of NaOH by reaction of formaldehyde with sodium sulfite to form sodium bisulfite-formaldehyde.

Reagents Required:

1. Sodium Sulfite, 1M; dissolve 125 g. anhydrous Na_2SO_3 in water & dilute to 1 liter solution. Prepare daily. Do not standardize.

2. Thymolphthalein Indicator, 0.1% in alcohol.

3. HCl, 1 N; See Standardization Procedure ~~0078~~ 9B

Procedure:

Measure 100 ml. 1M sodium sulfite solution into 250 ml. beaker with stirrer bar. ~~Add 3 drops thymolphthalein indicator &~~ Titrate to ~~just colorless~~ pH 10.0 with 1 N HCl solution. The quantity used should be <1 ml. (It is not noted.)

Weigh ~2 g. formaldehyde accurately in 250 ml. beaker & add 50 ml. distilled water. Add 3 drops thymolphthalein, & neutralize solution with 1 N HCl until just colorless or, if colorless initially, with 1 N NaOH until just blue, followed by 1 N HCl until colorless. If sample is strongly acid, use 50% NaOH for neutralization.

Add neutralized sodium sulfite solution to sample solution. Do not rinse beaker. Titrate resultant blue solution with 1 N HCl until colorless. Note quantity of HCl used in titration.

Calculation:

$$\% \text{ Formaldehyde} = \frac{(\text{ml. 1 N HCl})(\text{normality})(3.003)}{\text{wt. sample in g.}}$$

THIS ISSUE SUPERSEDES SAP 0078B DATED JANUARY 15, 1969.

RWK/smo

HAMPSHIRE CHEMICAL DIVISION

OF

W. R. GRACE & CO.

Standard Analytical Procedure

Number: 0101A

Date Issued: October 13, 1966

H_2SO_4 ASSAY

Principle of Method: Titration of H_2SO_4 with NaOH to Methyl Orange End Poin

Reagent Required:

Methyl Orange Indicator

.5N Sodium Hydroxide (NaOH): For preparation and standardization see Standardization Procedure No. 8

Procedure:

Accurately weigh out about 0.7 grams of the acid into a tared 50 ml. beaker and weigh. Wash the sample into a 250 ml. flask containing about 100 ml. of water.

Add several drops of methyl orange indicator.

Titrate the sample with .5N NaOH to an orange end point

Circulation:

$$\% H_2SO_4 = \frac{(\text{ml .5N NaOH})\ (N)\ (.049)\ (100)}{\text{Wgt. of sample}}$$

11/23/66 lcr

ORGANIC CHEMICALS DIVISION

HAMPSHIRE CHEMICAL

STANDARD ANALYTICAL PROCEDURE

Number: 0099D Date: September 10, 1975

Hydroxides as Na_2O in 50% NaOH; KOH in Caustic Potash

Principle of Method:

Carbonate is masked with neutral Barium solution; hydroxides are then titrated to a phenolphthalein endpoint.

Reagents Required:

1. Barium Chloride, 2% aqueous
2. Phenolphthalein, 1%
3. Hydrochloric Acid, 1.0N (see Standardization Procedure #9)

Procedure:

Weigh accurately 1.6-1.7 g. of NaOH or 2.5-2.6 g. KOH into a 250 ml. beaker. Dilute to 100 ml. with distilled water. Pour about 10 ml. of 2% $BaCl_2$ into a 50 ml. beaker and add 1 drop of phenolphthalein. Add 0.1N NaOH dropwise until color disappears. Pour the neutral barium solution into the sample beaker and titrate to the disappearance of the pink color with 1.0N HCl.

Calculations:

$$\% Na_2O = \frac{(\text{ml. HCl})(\text{normality})(3.1)}{\text{sample weight in g.}}$$

$$\% NaOH = (1.2906)(\% Na_2O)$$

$$\% KOH = \frac{(\text{ml. HCl})(\text{normality})(5.61)}{\text{sample weight in g.}}$$

THIS ISSUE SUPERSEDES SAP0099C DATED NOVEMBER 6, 1974.

RWK/smo

EXHIBIT G

Nitroparaffin Derivatives Specification Test Procedures

NPDST No.	Title of Procedure
1	Assay of Dry TA by Titration
2	Assay of 2-AB and AMP, and Determination of NMAB and AMP in 2AB by GC
3	Loss on Drying of TA
4	Melting Point of Dried TA
5	Determination of APHA Color of 2-AB, AMP, and a 20% Solution of TA
6	Determination of Water in 2-AB and AMP
7	Determination of Specific Gravity of 2-AB
8	Test for Heavy Metals in TA

ORGANIC CHEMICALS DIVISION

W. R. Grace & Co.

Nitroparaffin Derivatives Specification Test

Number:	NPDST 1	Date Issued:	
Title:	Assay of Dry TA by Titration		
Written by:	B. J. Ferdinand	Date:	2/8/89

Brief

Percent active ingredient of previously dried TA [tris(Hydroxymethyl)aminomethane] is determined by titration with 0.1 N HCl to a bromocresol purple endpoint.

Reference

USP XXI, Tromethane monograph.

Reagents and Equipment Required

- National Bureau of Standards TA reference material, 100%; dried by NPDST 3.
- 0.1 N HCl
- Bromocresol purple indicator crystals.
- 20 ml burette.
- Analytical 4 place balance.

Solutions Required

0.1 N HCl: Prepare according to Dilut-it or Accuit instructions or by weighing ~10 grams of 36.9% HCl to a liter volumetric and diluting to volume with distilled water.

Bromocresol Purple TS: Dissolve 250 milligrams bromocresol purple in 20 milliliters of 0.05N sodium hydroxide, and volumetrically dilute to 250 milliliters with distilled water.

Standardization of 0.1 N HCl

Dissolve ~ 250 milligrams of previously dried NBS-TA accurately weighed to ±0.1 milligram, in 100 milliliters of distilled water. Add 3-5 drops bromocresol purple TS. Titrate with 0.1 N HCl to a yellow endpoint. Repeat in triplicate. Calculate the normality of HCl by the following equation:

$$N_{HCl} = \frac{(\text{milligrams TA})}{(\text{milliliters HCl})(121.14)}$$

$$MW_{TA} = 121.14$$

Use the average N_{HCl} of the three titrations in the calculation below.

Assay of TA

Dissolve ~250 milligrams previously dried TA test sample (NPDST 3), accurately weighed to ±0.1 milligrams, in 100 milliliters of distilled water. Add 3-5 drops bromocresol purple TS. Titrate with standardized 0.1 N HCl to a yellow endpoint. It is recommended this titration be done in duplicate.

Calculate the percent active ingredient by the following equation:

$$\%AI_{(TA)} = \frac{(N_{HCl})\,(\text{milliliters HCl})\,(121.14)}{\text{milligrams of TA test sample}} \quad (100)$$

ORGANIC CHEMICALS DIVISION

W. R. Grace & Co.

Nitroparaffin Derivatives Specification Test

Number: NPDST 2 Date Issued:

Title: Assay of 2-AB and AMP, and Determination of NMAB and AMP in 2-AB by GC

Written by: W. M. Coleman Date: 2/2/89

Brief

Aminoalcohol test samples are dissolved in methanol before injection into a gas chromatograph by auto-injector. Components are separated by a capillary GC column and detected with a flame ionization detector. The amounts of components are determined by comparing peak areas with standards of known concentration using the external method of calibration.

Safety

Refer to MSDS sheets.

Apparatus

- Gas Chromatograph Hewlett Packard 5890A with FID, temperature programming, and split/splitless capillary column injector.
- Hewlett Packard 3393A computing integrator.
- Hewlett Packard 7673A auto-injector.
- Sample vials, septa, crimper available from Hewlett Packard Co.
- Cyanopropilphenyl (7%) 15 mtrs. x 0.53 mm id, 1µm film, DB-1701 megabore GC column. Available from J&W Scientific Inc., Catalog #1250712.
- Analytical balance with an accuracy of 0.1 mg.

Reagents

- 2-AB Standard
- AMP Standard
- N-methylaminobutanol Standard
- Methanol solvent (B&J) UV grade
- Hydrogen and air for FID
- Helium for carrier gas

Note: All standards and their certificates will be supplied by W. R. Grace

Conditions of Analysis

Column Temperature:	60°C
Initial hold time:	5 minutes
Program rate:	12°C/minute
Final temperature:	180°C
Helium flow:	2 psig
Split vent flow rate:	95 mls/minute
Injector temperature:	245°C
Detector temperature:	270°C
Auto injector:	see attached conditions

Standard Preparation

2AB Weigh ~5 gms 2-AB, ~0.05 gms NMAB, and ~0.015 gms AMP standards to a 100 ml volumetric flask. Record each weight to ±0.001 gms. Dilute to volume with methanol and mix well.

AMP Weigh ~5 gms AMP standard to a 100 ml volumetric flask and record the weight to ±0.001 gms. Dilute to volume with methanol and mix well.

Sample Preparation

Weigh ~5 gms of test sample to a 100 ml volumetric flask and record the weight to ±0.001 gms. Dilute to volume with methanol and mix well.

Procedure

Transfer sample and standard solutions to auto-injector vials for analysis. Inject each vial twice.

Note: Make sure area counts of the duplicate injections are reproducible before proceeding. Change septum daily to insure good reproducibility.

GC scans are attached.

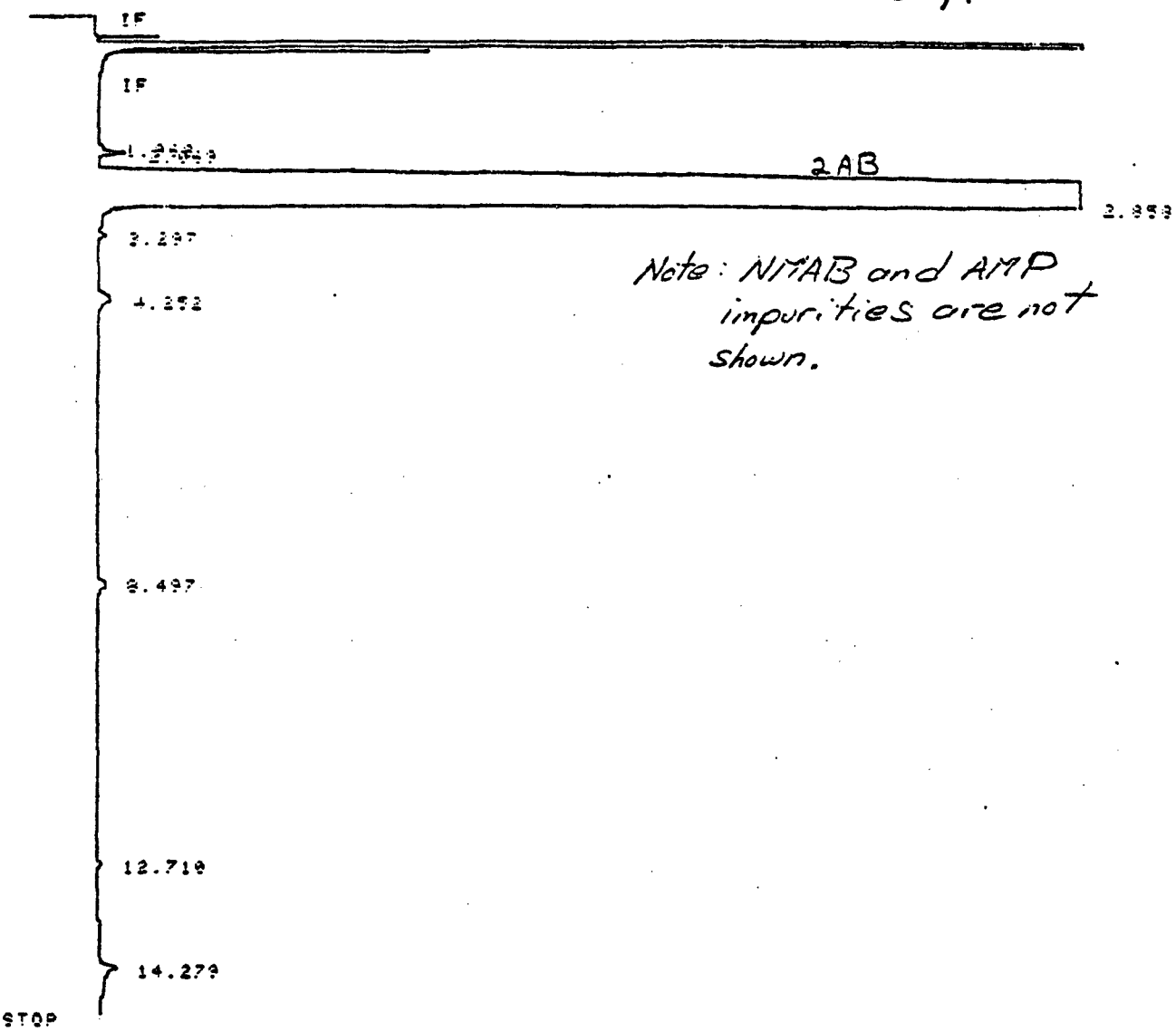
Calculations

$$\frac{\text{(avg.) area count component}}{\text{(avg.) area count standard}} \times \frac{\text{AI of standard}}{\text{weight of sample}} \times \text{weight of standard} = \text{weight \% of component of interest}$$

```
7673A SAMPLER:
 LOOP ADDRESS:  0

 REAR INJECTOR
  INJ/BOTTLE          2 -->     0
  FIRST BOTTLE       11 -->   1  0
  LAST BOTTLE        15 -->     0
  # OF SAMPLE WASHES  3 -->     0
  # OF PUMPS          6 -->     0
  VISCOSITY           0 -->     0
  VOLUME              1 -->     0
  # OF SOLVENT A WASHES  4 -->      0
  # OF SOLVENT B WASHES  0 -->      0
  PRIORITY SAMPLE (1=YES)  0 -->      0
  CAPILLARY ON-COLUMN  0 -->      0

SECTION TO BE EDITED:    BREAK

*  SEQ  START
```

2-AB standard
8.626 g/100

RUN # 657 JAN 31, 1989 14:53:58
START



Note: NITAB and AMP impurities are not shown.

STOP

RUN# 657 JAN 31, 1989 14:53:59

SAMPLE NAME: : SAMPLE# 2
NITRILE ANALYSIS-HRC PROCESS

../DERIVATIVES ANALYSIS

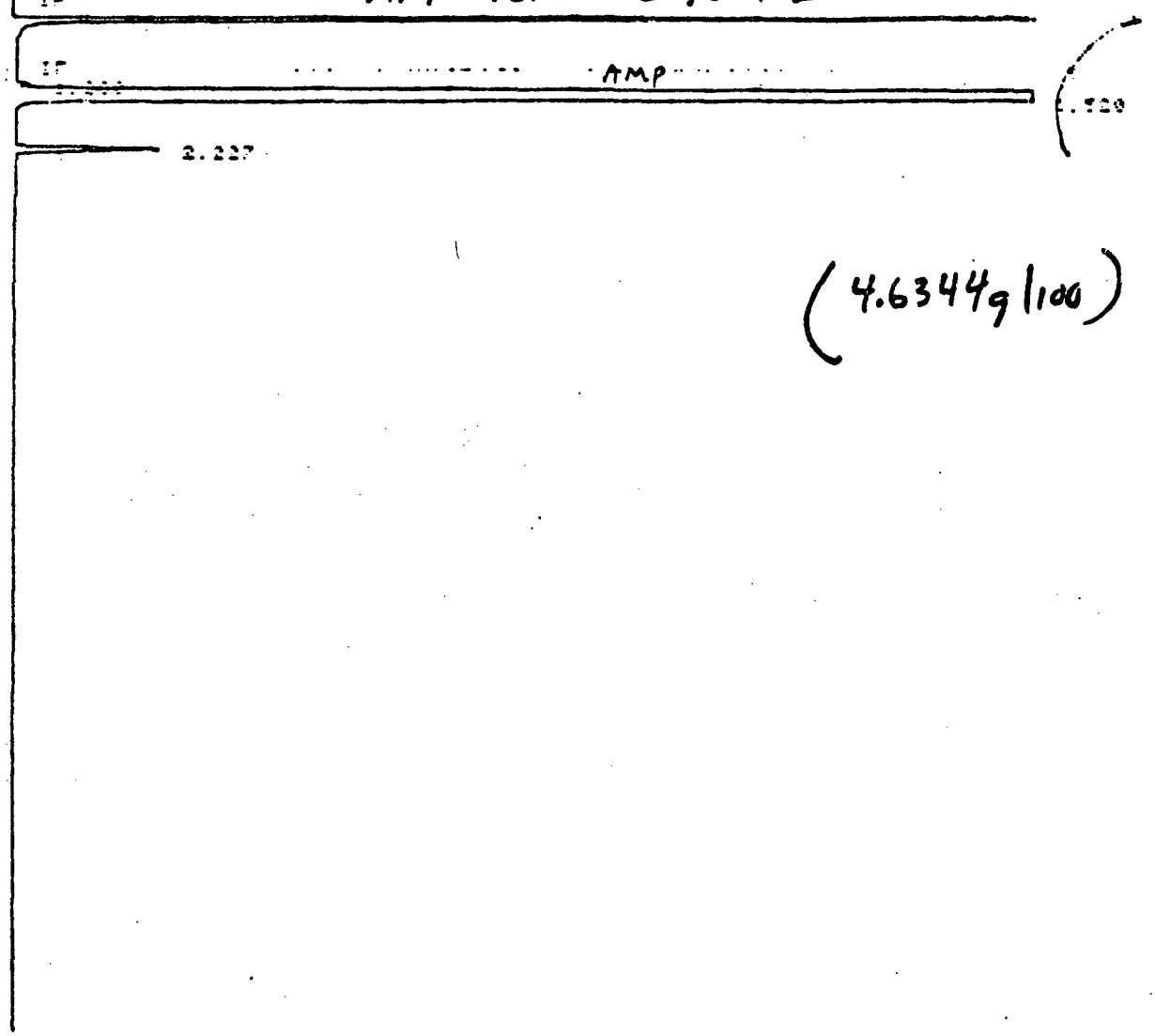
AREA%

RT	AREA	TYPE	WIDTH	AREA%
1.968	12973	PV	.073	.04399
2.069	48600	VB	.059	.16443
2.953	29272640	PV	.308	99.04131
3.297	22404	VB	.094	.07580
4.252	87654	PB	.195	.29657
9.497	13093	BV	.065	.04123
12.710	13205	BB	.091	.04469
14.279	80433	PV	.173	.27210

RUN # 661 JAN 31, 1989 16:15:30

START

AMP STd RJB 726-124



(4.6344g/100)

STOP

RUN# 661 JAN 31, 1989 16:15:30

SAMPLE NAME: ' ' SAMPLE# 6

NITRILE ANALYSIS-WRC PROCESS

1 ' ' DERIVATIVES ANALYSIS

T= 1320.2

AREA%

RT	AREA	TYPE	WIDTH	AREA%
1.290	18242	PV	.046	.13798
1.520	12960256	VB	.105	98.02947
2.227	242274	PB	.057	1.83253

TOTAL AREA=1.3221E-07

MUL FACTOR=1.0000E-00

ORGANIC CHEMICALS DIVISION

W. R. Grace & Co.

Nitroparaffin Derivatives Specification Test

Number:	NPDST 3	Date Issued:	
Title:	Loss on Drying of TA		
Written by:	B. J. Ferdinand	Date:	2/8/89

Brief

The percent weight loss of TA [tris(Hydroxymethyl) aminomethane] test sample dried at 105°C for 3 hours is determined.

Equipment Required

- Analytical 4 place balance.
- 5.5 cm x 1.5 cm glass petri dishes.
- Oven capable of 105°C.
- Dessicator.

Procedure

Tare a clean, dry glass petri dish, record its weight to ±0.1 mg (A), add ~1 gram TA crystals, record weight to ±0.1 mg (B). Repeat a duplicate of the sample. Place in 105°C oven for 3 hours. Remove petri dishes to a dessicator; cool for ~20 minutes. Weigh each dish, recording weight to ±0.1 mg (C). Calculate loss on drying by the following equation:

A: Weight tare
B: Weight tare + weight wet sample
C: Weight tare + weight dry sample

Weight wet sample = B - A
Weight dry sample = C - A

$$\text{Loss on drying} = \frac{(\text{weight wet sample}) - (\text{weight dry sample})}{\text{weight wet sample}} \times 100$$

Take a mean of the two samples. Results should be less than 1%.

ORGANIC CHEMICALS DIVISION

W. R. Grace & Co.

Nitroparaffin Derivatives Specification Test

Number:	NPDST 4	Date Issued:	
Title:	Melting Point of Dried TA		
Written by:	R. J. Bulka	Date:	2/9/89

Brief

Melting point is determined visually with a capillary melting point apparatus.

Reference

Refer to the melting point apparatus manual.

Equipment Needed (or equivalent)

- Hoover capillary melting point apparatus.
- Glass capillary tubes.

Procedure

The sample is dried at 105°C for 3 hours prior to analysis, as outlined in NPDST 3.

Load capillary tubes by pushing the open ends into the mass of dried crystals. Hold the tubes upright and tap or vibrate until the bottom 5-10 mm of the tubes are filled with crystals. Place the filled tubes in the melting point apparatus and begin heating.

Temperature can be increased rapidly until ~150°C, but beyond this point the rate of rise should be moderated to 1-2°C per minute. Temperature should be noted at the first sign of melting and at the moment when the last solid material has melted. Report melting point as the range between these two temperatures.

APHA Color Standards:

Prepared by serial dilution of 500 APHA Platinum Cobalt Color Standard (Fisher Scientific Cat. No. SP120).

Calibration:

The 500 APHA Pt/Co standard was diluted to prepare a set of color standards of 10, 20, 30, 40, 50, 100, 150, 250, 400, and 500 APHA. Two sets of color measurements were made:

(1) using the glass cell provided by Klett, 40 mm light path

(2) same as (1) but 20 mm light path

The raw data and the calibration curves for the same set of standards in the 2 cells are shown in the accompanying table and figures. Calibration factors were calculated in two ways:

Calculation:

(1) Graphically: the slope of the calibration curve = F

(2) A linear "best fit" equation was computed using the program "S-Stat".

These factors are:

	From Graphs APHA =	Calculated by X-Stat APHA =
for 20 mm cell	K x 2.71	[K x 2.98]-2
for 40 mm cell	K x 1.51	[K x 1.68]-2

where K = Klett reading

ORGANIC CHEMICALS DIVISION

W. R. Grace & Co.

Nitroparaffin Derivatives Specification Test

Number: NPDST 5 Date Issued:

Title: Determination of APHA Color of 2AB, AMP, and a 20% Solution of TA

Written by: W. M. Coleman Date: 2/6/89

Brief

The APHA color scale is a measurement of the "yellowness" of lightly colored solutions. Over the past 20 years, whenever we have measured visible spectra of yellow plant products, we have always found a very broad absorption band, seldom with an absorption maximum, with lowest absorption at ~500 nm, rising to maximum at the visible limit of the photocell, or 380 - 400 nm.

About 10 years ago we found that a simple filter photometer with a blue filter gave a response linear to the APHA color scale. Since then, all APHA measurements of Hampshire products have been obtained using a modified Klett colorimeter. We are using an unmodified Klett to measure APHA color of NP samples. This memo describes the exact procedure we are using.

Safety:

Refer to MSDS sheets.

Instrument:

Klett-Summerson Photolectric Colorimeter, Model 900-3; with matched pairs of 20 x 40 mm glass cells and #42 blue filter. Plugged into a voltage regulator (to minimize drift).

Procedure for Color Measurement of Samples

Because the region of interest is APHA ≤100, the maximum sensitivity is needed; therefore, color measurements are made using the 40 mm path length cell.

1. The instrument is adjusted to give a zero reading with dist. water in the pair of cells.

2. One cell is washed with dist. water and dried with acetone between readings. The other cell remains filled with dist. water and is used to zero the instrument before each reading. The other cell is filled with the sample to be measured (prefiltered if any particulate matter is present). The Klett color is recorded.

   TA is dissolved in d.w. prior to analysis (20% wt/volume).

   AMP and 2AB are measured NEAT.

   Note: The Klett has a 0-1000 scale. This reading x 0.002 = optical density or Absorbance.

3. The APHA color equivalent to the Klett reading is calculated. (We are using the factor calculated by X-Stat.)

   APHA color = Klett color x F - [y intercept]

   Example: A sample gives a Klett reading of 100 using the 40 mm cell. APHA color = [1.68 x 100] - 2 = 166.

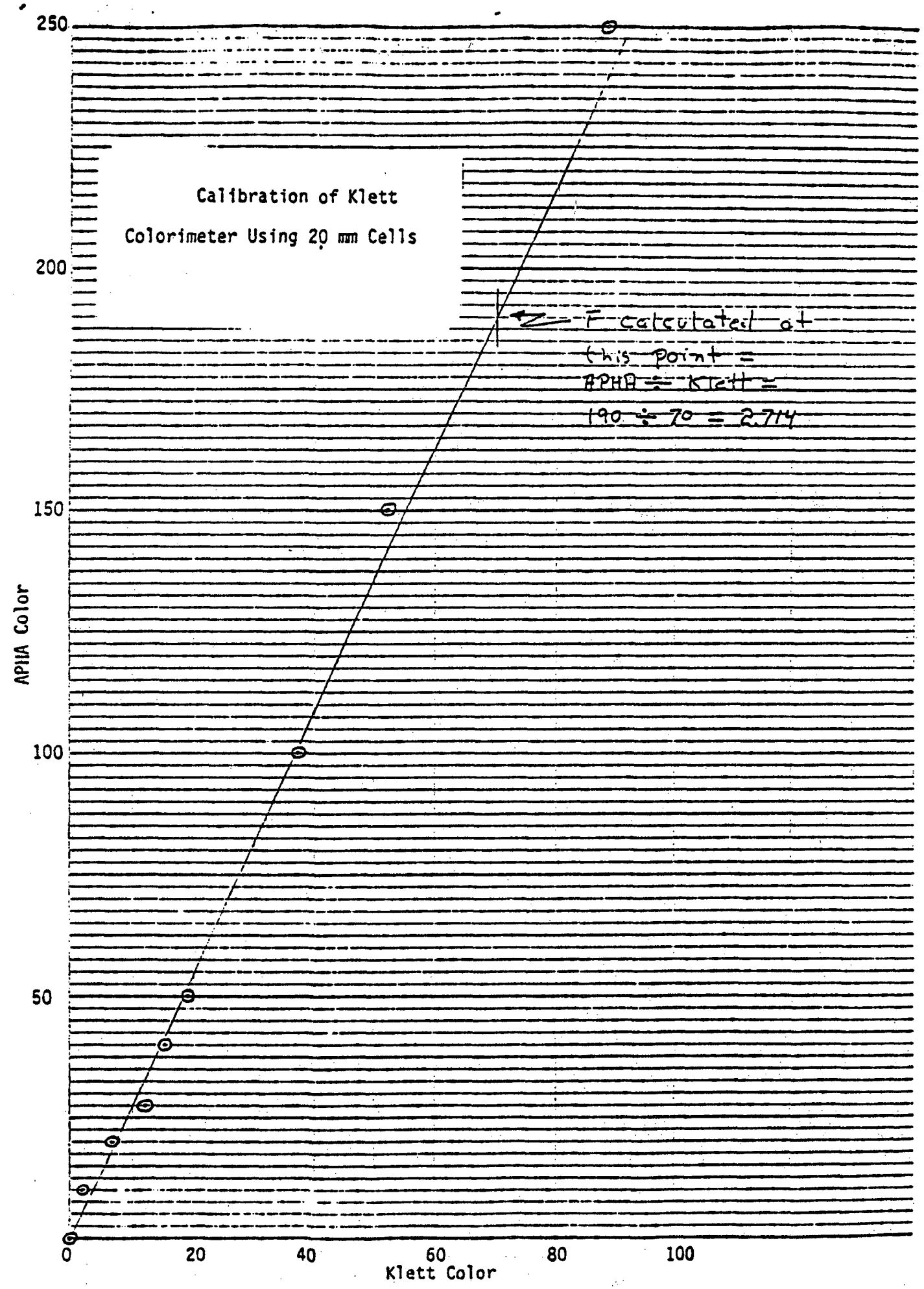
Klett Colorimeter Calibration Data

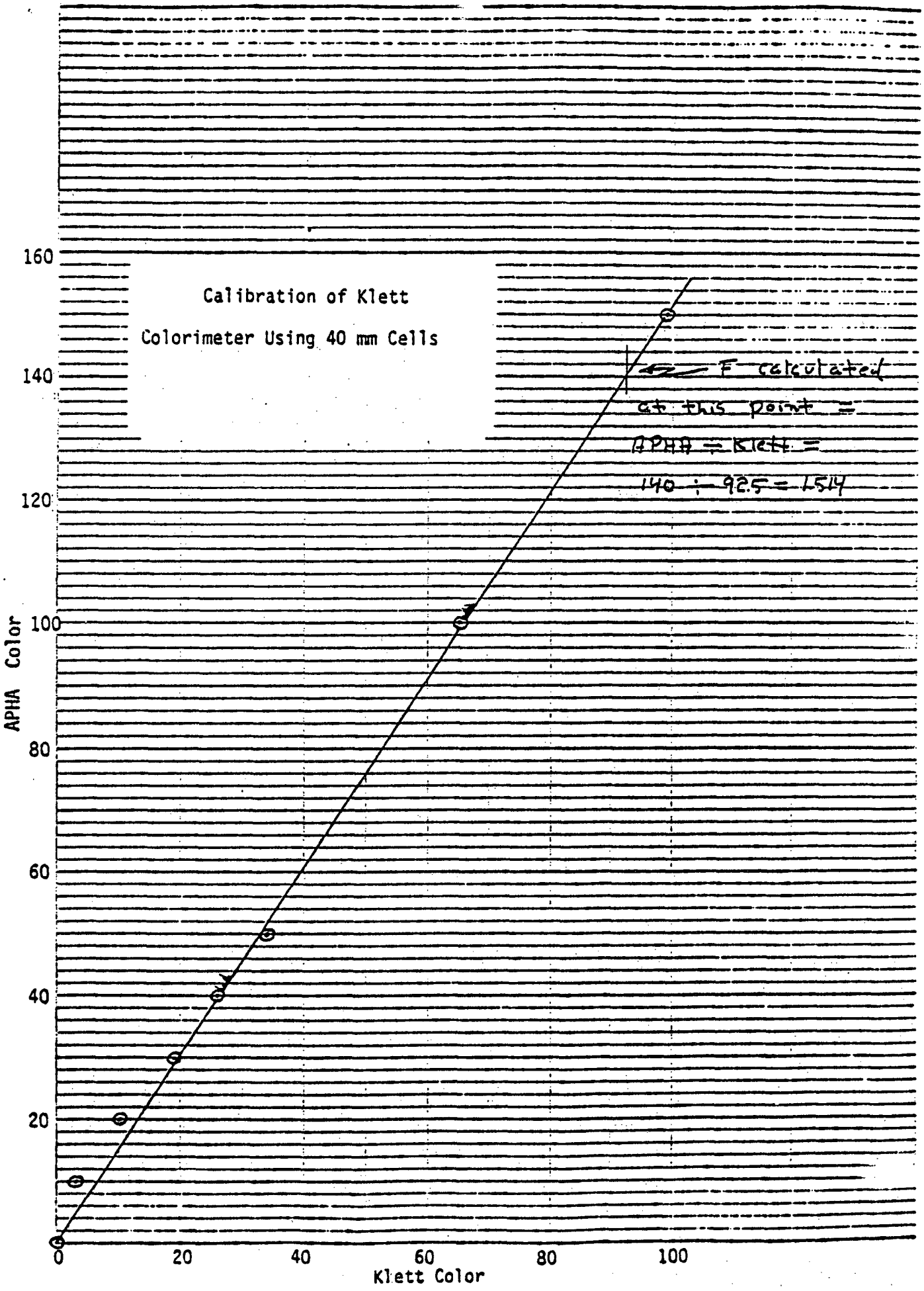
Preparation of Color Standards		Klett Readings	
ml 500 APHA Std Dltd to 50 ml	APHA Color	40 mm Cell	20 mm Cell
50	500	290	166
40	400	240	134
25	250	157	88
15	150	99	52
10	100	65	37
5	50	34	19
4	40	26	15
3	30	19	12
2	20	10	6.5
1	10	3	2
0	0	0	0

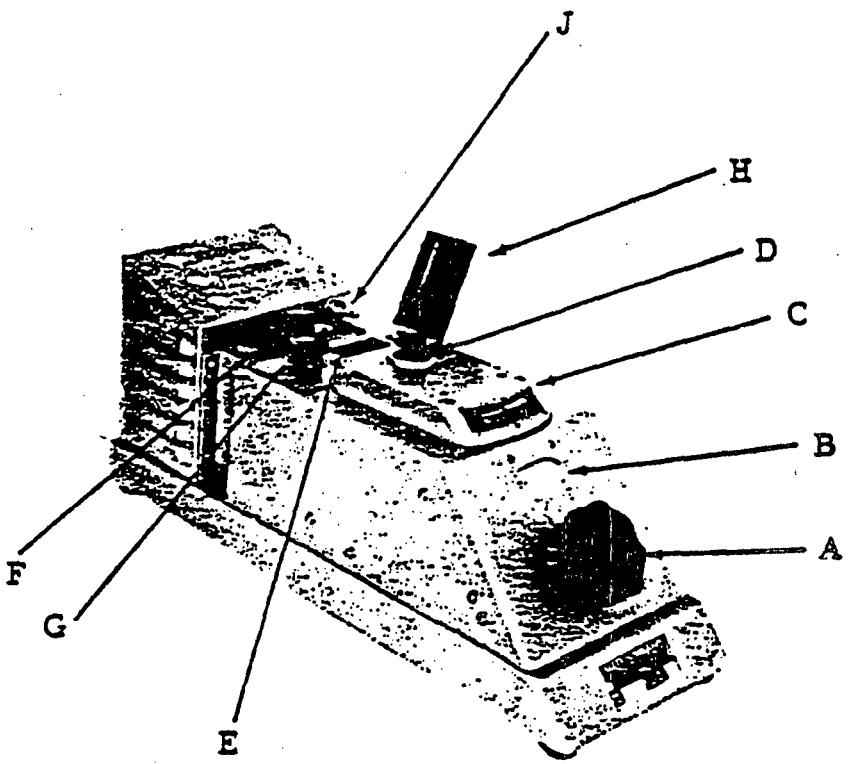
Ref: Memo from J. C. Thunberg dated 20 August 1987 to NP file.

Klett Colorimeter Calibration Data

Preparation of Color Standards		Klett Readings		
ml 500 APHA Std Dltd to 50 ml	APHA Color	40 mm Cell	20 mm Cell	10 mm Cell
50	500	290	166	80
40	400	240	134	62
25	250	157	88	38
15	150	99	52	26
10	100	65	37	18
5	50	34	19	6
4	40	26	15	7
3	30	19	12	4
2	20	10	6.5	3
1	10	3	2	2
0	0	0	0	0




Calibration of Klett
Colorimeter Using 40 mm Cells
F calculated
at this point =
APHA ÷ Klett =
140 ÷ 92.5 = 1.514
APHA Color
Klett Color
160
140
120
100
80
60
40
20
0
20
40
60
80
100



A – Scale Knob (Potentiometer dial)
B – Scale Reading (Potentiometer scale)
C – Pointer (galvanometer)
D – Galvonometer pointer adjustment
E – Glass Cell
F – Filter Holder
G – Zero Adjustment Knob
H – Cell Compartment Cover
J – Light Switch

ORGANIC CHEMICALS DIVISION

W. R. Grace & Co.

Nitroparaffin Derivatives Specification Test

Number:	NPDST 6	Date Issued:
Title:	Determination of Water in 2-AB and AMP	
Written by:	J. N. LePage	Date:

Brief

Water is determined in 2-AB and AMP by the Karl Fischer method. For the titration system used in this procedure, acetic acid must be added to the titration vessel prior to analysis of these basic substances.

References

Refer to the Karl Fischer titrator manual.

Equipment Needed (or equivalent)

- Quintel Corporation "Computrac MS-1" Karl Fischer titrator.
- Four place balance.

Reagents Needed (or equivalent)

- Quintel "Single Solution Karl Fischer Reagent" Cat. No. 5305.
- Quintel "Methanol Free Solvent" Cat. No. 5325.
- Hydranal Sodium Tartrate-2-Hydrate Standard (contains 15.66 ± 0.05% H_2O).
- Reagent Grade Glacial Acetic Acid.

Procedure

Set up the Karl Fischer titrator as recommended in the operators manual. To the 15-20 mls of the "Methanol Free Solvent" add ~ 5 mls of acetic acid to the titration cup. Instrument settings: Mode 1 and 30 second Wait Time.

Standardization

To a clean dry test tube add about 300 mg of sodium tartrate dihydrate standard. Accurately record the weight of the test tube and its contents to ± 0.1 mg (W_{T+S})

To the "blanked out" acidic KF titration cup add the dihydrate standard from the test tube, being careful not to spill the standard anywhere except into the titration solvent. Be careful not to contaminate the test tube with liquid or grease from the titration cup. (Note: some hydrate standard is expected to remain in the test tube.) Start the KF titrator. Record the mls of KF reagent used to reach the endpoint. Reweigh the test tube that contained the standard and record its weight to ±0.1 mg (W_T)

KF Titer Calculation

The amount of hydrate standard used for the standardization is determined by the weight difference of the test tube with standard (W_{T+S}) and without standard (W_T).

Calculate the titer of the KF reagent as follows

$$\text{KF titer in } \frac{\text{mg } H_2O}{\text{ml reagent}} = \frac{(W_{T+S}-W_T) \times 0.1566^*}{(\text{mls KF reagent})}$$

Duplicate standardizations should agree within 0.5% relative of each other.

*(Note: the actual fraction of water listed on the hydrate standard container should be used in the titer calculation.)

Sample Analysis

Using a disposable plastic eye dropper draw up an appropriate amount of test sample. (For 2-AB use ~1.5-2 mls, for AMP use ~0.5 mls.) Weigh the eye dropper containing the test sample and record the weight in grams to ±1 mg (W_{E+s}).

To the "blanked out" acidic titration cup add the test sample in the eye dropper being careful not to spill the sample anywhere except into the titration solvent. Be careful not to contaminate the eye dropper with liquid or grease from the titration cup. Start the KF titrator. Record the mls of KF reagent used to reach the endpoint. Reweigh the eye dropper that contained the test sample and record its weight in grams to ±1 mg (W_E).

$$\%H_2O = \frac{(\text{KF titer})(\text{mls of KF at endpoint})}{(W_{E+s}-W_E)} \times 10$$

This analysis should be rerun with a fresh charge of KF solvent containing ~5 mls of acetic acid if test results are higher than expected or if the titrator takes longer than usual to reach the endpoint. See discussion.

Discussion

Acetic acid must be added to the titration cup of the Computrac MS-1 to neutralize the basicity of the amine test sample. If the basicity of the amine is not neutralized, the Computrac MS-1 will give falsely high results. 5 mls of acetic acid is sufficient to neutralize ~3g of total test sample added to the cup before a fresh charge of KF solvent-acetic acid must be charged to the titration cup. A suspiciously high water result or a longer than usual time for the titrator to reach the endpoint may indicate the titration media is too basic. A fresh charge of KF solvent with acetic acid should then replenish the spent titration media.

Other KF titrators-solvent systems may not experience the problem described above.

ORGANIC CHEMICALS DIVISION

W. R. Grace & Co.

Nitroparaffin Derivatives Specification Test

Number:	NPDST 7	Date Issued:	
Title:	Determination of Specific Gravity of 2-AB		
Written by:	J. N. LePage	Date:	2/13/89

Brief

The density of 2-AB test samples is determined at 20°C. The specific gravity is calculated from the density.

Experiment Required

- KIMAX 110 ml graduated volumetric flask, Class A, Cassia (VWR Cat. No. 29630-000). This flask is graduated in 0.1 ml increments from 100 to 110 mls at 20.0°C.
- Circulating water bath set at 20.0°C.
- Funnel with a narrow ~10 cm long stem.
- Balance capable of 200g capacity with ± 0.1g accuracy.

Procedure

Weigh a dry stoppered 110 ml graduated volumetric flask, record the weight to ±0.1g (W_F). With the aid of a funnel, add ~105 mls of 2-AB test sample to the graduated volumetric flask. Carefully remove the funnel to prevent wetting the ground glass joint with test sample.

Weigh the stoppered flask containing the test sample. Record the weight to ±0.1g (W_{F+S}).

Place the stoppered flask in a 20°C water bath. The water bath level should be above the level of the test sample in the flask. After 2 hours, read the volume of the test sample in the flask to ±0.1 ml (V_S).

Calculation

Calculate the density of the test sample:

$$\text{Density}_{TS} = \frac{(W_{F+S})-(W_F)}{(V_S)}$$

Calculate the specific gravity of the test sample:

$$\text{Specific Gravity}_{TS} = \frac{\text{Density}_{TS}}{\text{Density of water at 20°C}}$$

$$= \frac{\text{Density}_{TS}}{0.998}$$

ORGANIC CHEMICALS DIVISION

W. R. Grace & Co.

Nitroparaffin Derivatives Specification Test

Number:	NPDST 8	Date Issued:	
Title:	Test for Heavy Metals in TA		
Written by:	J. N. LePage	Date:	2/13/89

Brief

2.0 gms of test sample is digested with sulfuric and nitric acids. A slightly acidic solution of the clear digest is treated with H_2S water. The metal sulfide color of the test sample is less than the color of a solution containing 20µg of lead standard.

This procedure recommends the use of a Hellige Aqua Tester (or similar device) to compare the color of the test sample with the lead standard solution. Matched color comparison tubes can also be used for comparing the color intensities without special viewing equipment.

Safety

Hydrogen sulfide (H_2S) is a highly poisonous gas. Handling of H_2S gas or solutions containing H_sS should be done in a well ventilated hood.

Refer to MSDS sheets for guidelines on the proper handling of chemicals used in this procedure.

Equipment Required

- Hellige Aqua Tester, Hellige Inc., Garden City, N.Y.
- Hellige Nessler Tubes, No. 611-T (Thomas Scientific).
- No. 3130 Biocolorimeter tube, 40 ml capacity. Mark the tubes at the 30 ml volume level.
- Crucible, VYCOR® brand, 30ml capacity, with covers.
- Platinum tipped tongs.
- Muffle furnace capable of 500-600°C temperature.
- 500 ml amber bottle with stopper.
- Bunsen burner with tripod and crucible triangle.

- Hot Plate.
- Red litmus paper.
- pH 3-4 short range pH indicator paper.

Reagents Required

- Concentrated sulfuric acid.
- Concentrated nitric acid.
- ~6N HCl (dilute ~equal volumes of water and concentrated hydrochloric acid).
- ~6N aqueous ammonia (dilute ~1 part conc. NH_3 with 2 parts water).
- ~1N acetic acid (dilute ~60 grams of glacial acidic acid with water to make 1 liter).
- Lecture bottle of hydrogen sulfide.
- Lead nitrate.

Special Solutions

Lead (Pb), 100µg/ml Standard Solution:

Add 159 ±1 mg of $Pb(NO_3)_2$ to a 100 ml volumetric flask. Add ~1 ml of conc. HNO_3 and dilute with H_2O to make 100 mls of solution. (Prepare fresh every three months.)

Pb, 10µg/ml Standard Solution: Dilute 10 mls of Pb 100µg/ml Standard Solution with water to make 100 mls of solution. (Prepare fresh weekly.)

H_2S Water: Fill a 500 ml amber bottle with cooled distilled water, leaving a minimum of headspace in the bottle. Slowly bubble H_2S gas into the water for 2-3 minutes. Securely cap the H_2S water bottle and store in a refrigerator. The solution should be made up to volume with water and resaturated with H_2S weekly.

Sample Preparation

Weigh 2.0g of TA test sample to a clean crucible. Dropwise, slowly add conc. H_2SO_4 to the crucible to wet the sample. Carefully and thoroughly char the sample with a low flame. Cool the crucible. Add ~2 mls of conc. HNO_3 and ~5 drops of conc. H_2SO_4 to the charred material. Heat over a low flame, with increasing temperature until white fumes are no longer evolved. Place the crucible in a muffle furnace set at 500-600°C for ~2 hours The test sample residue should now be nearly colorless. (Repeat flame and muffle treatment with a mixture of ~2 mls of nitric and ~5 drops of H_2SO_4 if the residue has substantial color.) Cool the crucible.

Wash down the inside of the crucible with ~4 mls of ~6N HCl. Cover the crucible and slowly heat for ~15 minutes on a hot plate. Remove the cover and continue heating until just dry. Add ~2 drops of ~6N HCl and ~10 mls of water and heat for about 10 minutes, then cool. Add more H_2O to maintain ~10 mls of solution in the crucible.

Dropwise add ~6N NH_3 to the crucible until the solution is just basic to red litmus paper. Add water to make ~25 mls of solution, and adjust to pH 3-4 with ~1N acetic acid using short range pH indicator paper.

Procedure:

Standard Tube: Add ~25 mls of H_2O to a ~40 ml color comparison tube. Pipet 2.0 mls of Pb 10µg/ml Standard Solution (20µg Pb) to the tube. Dropwise add ~6N ammonia or ~1N acetic acid to the tube and adjust to pH 3-4 using short range pH indicator paper. Add additional water to make 30 mls of solution.

Test Sample Tube: Add the digested test sample crucible contents to a ~40 ml color comparison tube. Wash the inside of the crucible with several ml portions of H_2O. Add the washings to the tube to make 30 mls of solution. This solution should be colorless and free of particulate matter.

To standard and test sample color comparison tubes add 10 mls of H_2S water, mix, and allow the solutions to stand for ~5 minutes.

Place the standard and test sample tubes into the Hellige Aqua Tester and compare the color intensities of the solutions. The test sample solution should not be darker than the Pb standard solution.

GRACE
Organic Chemicals Division

NPAP-27

NITROPARAFFINS

ANALYTICAL PROCEDURE

NUMBER: NPAP-27

TITLE: GC Analysis of 1-Nitropropane and 2-Nitropropane

ISSUE NO.: 3

DATE OF ISSUE: [redacted]

REASON FOR REISSUE: Update Method

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1. INTRODUCTION

The assay of 1-Nitropropane and 2-Nitropropane is easily obtained by gas chromatography. The method of quantitation is internal standardization, using Butanol as the internal standard. In this procedure, the impurities are accurately quantitated, summed and subtracted from 100% to give the product assay. The detector used is a flame ionization detector.

2. SPECIAL PRECAUTIONS

Nitroparaffins are flammable and toxic. Skin contact and inhalation should be minimized. Caution, 2-Nitropropane is a possible carcinogen. As always, EYE PROTECTION IS TO BE WORN AT ALL TIMES!

3. INSTRUMENT AND CONDITIONS

Gas Chromatograph
Temperatures: injection port 240°C
column program initial 240°C, Isothermal, hold for 20 minutes.
Carrier Gas: Helium at 30 mls/min flow rate
Detector: Flame Ionization
Column: 8'X1/8" O.D. 316 Stainless Steel with 100/120 mesh Porapak Q or QS Packing

CEDAR CHEMICAL CORPORATION
P. O. Box 2749, Highway 242S.
West Helena, AR 72390
Phone: (501) 572-3701
Fax: (501) 572-3795

March 30, 1990

Mr. Richard C. Zagraniczny
W. R. Grace & Co.-Conn.
55 Hayden Avenue
Lexington, MA 02173

Re: Procedures for 2-Amino-2-Methyl-1-Propanol Waste Disposal

Cedar Chemical Corporation ("Cedar") agrees to practice the following procedures in the disposal of wastes generated from the manufacture of 2-Amino-2-Methyl-1-Propanol ("AmPro") for W. R. Grace & Co.-Conn. ("Grace").

a) Cedar will complete a waste manifest, a Certificate of Certification and a standard Bill of Lading to accompany each shipment of waste in conformance with all government regulations.

b) Cedar shall ship AmPro aqueous waste to Empak Inc.'s facility in Deer Park, Texas, using transport equipment provided by Trinity Chemical Industries. These will generally be 20,000 gallon railcars, although in the absence of railcars, tank truck shipment is acceptable. Cedar will ensure that the composition of the AmPro aqueous waste shall not exceed the maximum of the range indicated in the attached Generators Waste Profile. Any waste whose composition falls outside this range should not be shipped without prior approval from Grace.

c) Cedar shall ship AmPro aqueous waste containing nickel to Empak Inc.'s facility in Deer Park, Texas, using transport equipment provided by Trinity Chemical Industries. These will be rubber lined tank trucks. Cedar will ensure that the composition of the AmPro aqueous waste containing nickel shall not exceed the maximum of the range indicated in the attached Generators Waste Profile. Any waste whose composition falls outside this range should not be shipped without prior approval from Grace.

d) Cedar shall ship AmPro distillation bottoms to Rollins Environmental Services, Inc.'s facility in Baton Rouge, LA,

using whatever transportation equipment that is appropriate. Cedar will ensure that the composition of the AmPro distillation bottoms shall not exceed the maximum of the range indicated in the attached Generators Waste Profile. Any waste whose composition falls outside this range should not be shipped without prior approval from Grace. Grace may periodically instruct Cedar in writing to ship the AmPro distillation bottoms to an alternate off-site location.

e) Cedar shall ship spent Raney Nickel catalyst to an off-site location designated by Grace for recovery of nickel. Grace shall provide the name of the selected recycler in writing at a later date, as an amendment to this agreement.

f) Cedar shall dispose of all solid wastes other than spent Raney Nickel catalyst that will be generated by the manufacture of AmPro for Grace at a fully permitted Class I facility.

This constitutes the "Procedures" for the disposal of AmPro waste referred to by Articles 4(g) and 13.7 of the March 10, 1989 Agreement between Cedar and Grace for the manufacture of amino alcohols.

Sincerely,

J E Porter

Joe E. Porter
Environmental Engineer

JEP:doc

Attachment

Approvals:

John H. Miles
Cedar Chemical Corporation

Fred Huber 3/30/90

Fred Huber
W. R. Grace & Co.-Conn.
Organic Chemicals Division

Grace Contract

CEDAR CHEMICAL CORPORATION

24th Floor • 5100 Poplar Avenue • Memphis, TN 38137 • 901-685-5348

May 11, 1990

Mr. Richard C. Zagraniczny
Product Development Manager
W. R. Grace & Co.
Organic Chemicals Division
55 Hayden Ave.
Lexington, MA 02173

Dear Richard:

In accordance with our telephone conversation of today, enclosed are Change Orders allegedly requested by W.R. Grace relative to the Nitroparaffin Derivatives Project. These Change Orders were recently prepared by Jim Fowler of Delta Process Management, Inc. and never formally approved by either Grace or Cedar. However, as discussed, I would appreciate your reviewing them and receiving your comments at an early date.
Thank you.

Sincerely,

Bill

William J. Eissler, Jr.
Vice President & General Manager
Organic Chemicals

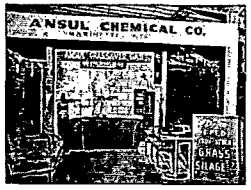
WJE/bd

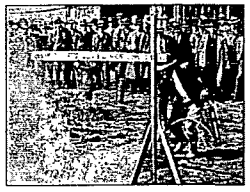
Enclosure

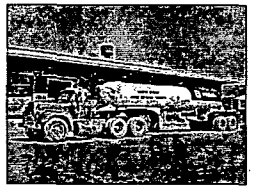
CC-J.R. Tomblin
G. Pratt
A. Malone

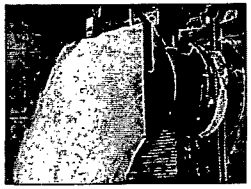
PIONEERS OF DRY CHEMICAL
ANSUL
REG. U.S. PAT. OFF.
FIRE EXTINGUISHING EQUIPMENT

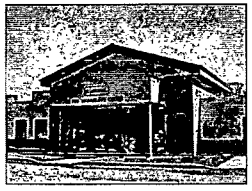
•1912 The Bastol Company is organized to make cattle feed out of sawdust. Great idea, but ahead of its time. Company goes bankrupt. **•1915** Frank Hood buys Bastol Company for its Sulfur Dioxide (SO_2) plant and renames "ANSUL" for **AN**hydrous **SUL**fur Dioxide. Starts selling SO_2 to die works and fruit preservers, and later as a refrigerant. **•1915-1938** The company capitalizes on mechanical refrigeration booms, grows and prospers right through the Great Depression. *President:* Francis G. Hood **•1915-1963** *Company Name:* Ansul Chemical Company. **•1927** New Sulfur Dioxide plant built. **•1931** Ansul Chemical Company of California formed. **•1934** Ansul forms part-time international sales department and begins to export products. **•1935** Research lab and pilot plant built. **•1936** To maintain its market position, Ansul builds a plant to produce a popular refrigerant: methyl chloride. **•1938** E.I. DuPont develops FREON, a new and better refrigerant. Ansul reacts by becoming a national distributor for FREON. Ansul starts an Industrial Chemicals division to find new markets for sulfur dioxide and methyl chloride. **•1938-1948** *President:* Harvey V. Higley **•1939** Refrigeration looks less and less like the business of the future for Ansul. Company buys DuGas Engineering Company, a small struggling manufacturer of dry chemical fire protection equipment. Ansul introduces first cartridge-operated fire extinguisher. DuGas brand is predecessor to today's RED LINE®. **•1940** Dry chemical is such a new idea that nobody understands how to use it. Ansul starts a fire school in Marinette to train employees. **•1942-1946** *Company Slogan:* "The Master of Flame" (duGas) **•1944** Fire protection assembly plant opens. **•1945** Ansul introduces first dry chemical trucks. **•1946-1948** Ansul introduces a completely redesigned line of fire extinguishers; uses its dry chemical know-how to develop new dry chemical extinguishing agents. **•1946-1949** *Company Slogan:* "The Master of Flame" (Ansul) **•1947** First large-scale fire tests in Marinette, Wisconsin. **•1948** The first Ansul Fire School to train customers. The school becomes *world famous and by 1987 will have* trained more than 40,000 firefighters. **•1948-1949** *President:* Francis J. Hood **•1948-1953** *Company Slogan:* "Pioneers of Dry Chemical" **•1949** Ansul introduces the first wax-free refrigeration oil. Then follows quickly with a line of SYSTEM BOSS filter dryers and DRY-EYE moisture indicators. **•1949-1974** *President:* Robert C. Hood **•1953-1955** *Company Slogan:* "Call the Ansul Man" **•1955** To finance future growth, Ansul "goes public," has its stock listed on the American Stock Exchange. Ansul Chemical Company of Venezuela formed. **•1956-1960** Due to rapid growth, Ansul can't cover the world with its own sales force so company establishes a nationwide distributor network for fire extinguishing products, systems and services. **•1959** Ansul becomes the exclusive national distributor of UCON for Union Carbide. Ansul International Corporation formed. **•1959-1961** *Company Slogan:* "New Products, New Ideas for Better Fire Protection" **•1960** Fire test station moves to Pierce Avenue (Marinette, Wisconsin). Ansul introduces first "Class D" dry powder agents for burning metals. Ansul introduces first dry chemical system to protect mobile mining equipment/vehicles. **•1961** Using its industrial savvy, Ansul goes into the agricultural chemical business; quickly becoming an important player in weed control and tobacco chemicals. For killing suckers on tobacco plants, Ansul offers SUCKER-PLUCKER®, SUPER STUFF®, and SUPER SUCKER®. It also offers BOLLS-EYE® as a cotton defoliant. Ansul Chemical Company of Mexico *established.* **•1962** *Ansul's future in the* refrigeration business looks bleak as the company tries to compete with giants like DuPont, Allied Chemical and Union Carbide. Ansul decides to exit the sulfur dioxide and methyl chloride business to focus solely on agrichemicals and fire protection. Ansul introduces R-100 dry chemical system for the protection of restaurant appliances, hoods, and ductwork. Ansul has been exporting since 1934, but senses need to manufacture overseas. Buys fire protection companies in Belgium (Protection Generale Incende) and Holland (Minimax). Is soon manufacturing in six foreign countries, selling its products in almost every country in the free world. **•1963** Ansul acquires Mason Electric Company (California) and establishes Ansul Chemical, Ltd. (England). **•1963-1981** *Company Name:* The Ansul Company **•1964** Ansul opens dry chemical plant in Oakville, Ontario, Canada and forest fire equipment plant in Couderay, Wisconsin. **•1965** New products include insect spray (dispensed through a modified MERRIMAC® fire extinguisher) and SILVISAR tree-killing system including a tree-killing chemical supplying a HYPO-HATCHET® injector. **•1967** Ansul sells refrigeration division. Weslaco (Texas) Development Center established. Ancon Chemical Co. established in Malaysia to make agricultural chemicals. Plant manufactures ANSAR® 529M, Malaysia's first herbicide. **•1968** Functional Services Center (now FX Building) built. Acquisition of Olin package. **•1969** Research center opens in Madison, Wisconsin. Acquisition of Turex. **•1970** Sierra Group (California) established. **•1971** Ansul buys one of Australia's largest chemical companies - Amalgamated Chemical - from Continental Oil *Company. The company inherits a large* agricultural market including products for rubber and oil palm, timber treatment, and snail and slug control. Ansul acquires Eagle River Chemical Company (Arkansas). **•1972** Ansul acquires Lane, Ltd. (Australia). **•1973** Ansul listed on the New York Stock Exchange. **•1974** Ansul acquires Dover Chemical (Ohio) and SOFRAMI (France). **•1974-1976** *President:* Morris L. Neuville **•1976** Ansul sells its entire ag chem business putting all of its energies behind fire protection. **•1976-1980** *President:* Terrell L. Ruhlman **•1977-1979** *Company Slogan:* "The Fire Protection Company" **•1978** Company is acquired by Wormald International, Australian based and the world's largest fire protection company. First issue of Burning Issues newsletter published. **•1979-1985** Ansul lives. Wormald wisely leaves the company more or less alone; and Ansul introduces new products, enters new markets, embarks on acquisition program and captures a leadership position in the U.S. fire protection market. **•1980-1983** *President:* William A. Rickel **•1981-1995** *Company Name:* Ansul Fire Protection **•1982** Ansul introduces R-102 wet chemical restaurant system for the protection of cooking equipment - appliances, hoods, and ductwork. **•1983-1986** *President:* Marc V. Gross **•1986** Ansul enters new market introducing a line of SPILL-X® products including agent and applicators for hazardous spills of acids, caustics, solvents, and formaldehyde. **•1986-1989** *Company Slogan:* "The First Name in Fire Protection" **•1987-1990** *President:* J. Donald Roland **•1988** Ansul introduces ANSULITE® 3x3, *the first alcohol-resistant AFFF capable* of being used on both hydrocarbon and polar solvent fuels at a 3% concentration. **•1989** *Company Slogan:* "Safeguarding Life & Property." Ansul introduces SILV-EX®, the first foam concentrate for "Class A" wildfires... later promoted for structures, tires, and paper. **•1989-1994** *Company Slogan:* "Safeguarding Life, Property and the Environment" **•1990** Wormald International is purchased by Tyco International, Ltd. solidifying the company's status of world's largest fire protection company. Ansul introduces INERGEN® inert gas, clean agent system as an alternative to Halon 1301 which was banned from production via the Montreal Protocol. **•1990-1992** *President:* Mark E. Mathisen **•1992-2002** *President:* Karl J. Kinkead **•1993** Ansul acquires Rockwood Foam to complete its full foam product line. **•1994-2003** *Company Slogan:* "Experts in Global Fire Solutions" **•1995** Ansul acquires Preferred CO_2 (Ohio) and markets Bulk and exclusive "Mini-Bulk" low pressure CO_2 storage technology. **•1995-** *Company Name:* Ansul Incorporated **•1997** Ansul introduces first "low viscosity" foam concentrates. **•1998** Ansul introduces PIRANHA® restaurant system featuring the first hybrid concept: wet chemical with water follow-up. Ansul acquires Pyro Technologies, Inc. (New Jersey) including various pre-engineered fire protection products under the PYRO-CHEM® brand name. Ansul introduces the K-GUARD® "Class K" *wet chemical fire extinguisher for* cooking equipment. **•1999** Ansul introduces twin-agent concept for non-road mobile equipment: dry/wet chemical discharge. **•2000** Ansul introduces TARGET-7® vapor mitigating/acid neutralizing agent for acid spills. **•2001** Ansul acquires Flag Fire Equipment of Ontario, Canada and introduces new fire extinguisher line to Pyro-Chem distributors. **•2002-** *President:* Mark VanDover **•2003-** *Company Slogan:* "Innovative Fire Solutions" **•2004** Ansul reintroduces MAGNUM™ rapid intervention twin-agent vehicles. **•2005** Ansul introduces CLEANGUARD® non-magnetic clean-agent extinguisher for MRI rooms **•2006** Ansul opens new Fire Technology Center housing the Ansul Fire School and other product training and demonstration facilities.











The next century... The next 100 years are going to be fun. No predictions, no guarantees, but we know we'll be here offering innovative fire solutions. After all, the company has had a lot of experience in meeting change head-on and adapting to it on a global basis.

ANSUL®
Innovative Fire Solutions

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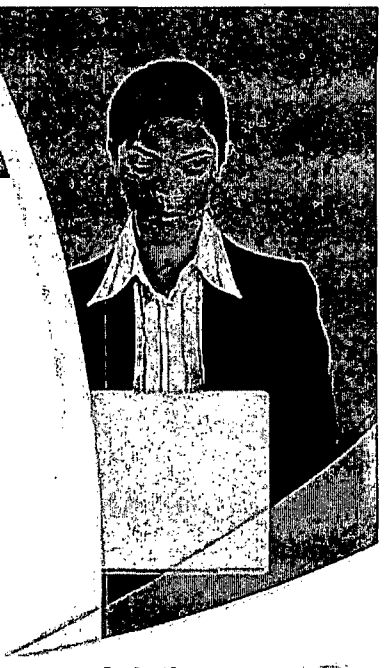
Our History



Every moment of every day Tyco is doing something vital, strengthening our company, helping our customers to succeed and improving the lives of people around the world.

Our history – over 120 years protecting life, property and the environment.
Our organisation was first created in 1845 under the name of Mather & Platt. By 1883 having acquired the manufacturing rights to the Grinnell sprinkler outside of the USA, our organisation developed a global fire protection company spanning the four continents of Europe, Asia, Africa and South America.

By 1889 Sir John Wormald of Mather & Platt enlisted the aid of his brothers in Australia to create a distributorship for the Grinnell sprinkler. The Wormald brothers went on to develop a fire protection empire of their own and in 1976 they acquired Mather & Platt, which had created them nearly 100 years before.

Wormald International Limited had grown by acquisition into a US$1 billion organisation by 1990 when it decided to join the Tyco family. This merger brought the entire world's foundling fire protection companies all back together again to become one truly global fire & security organisation spanning all five continents of the world.

When Arther J. Rosenburg, PhD founded Tyco in 1960 by opening a research laboratory for the purpose of carrying out experimental work for the government he could barely have imagined that the changes in focus the organisation made would provide thousands of vitally important products and services serving millions of customers in over 100 countries.

Our name was changed from Tyco Laboratories, Inc to Tyco International Ltd. in 1993 to reflect Tyco's truly global presence. In early 1998, ADT joined the Tyco family as part of its Fire and Safety Services Group.

Tyco International today
On 29th of June 2007, Tyco spun off its electronics and healthcare businesses into two independently publicly held companies. Tyco Electronics and Covidien (formerly Tyco Healthcare) now operate totally separately from Tyco, with their own board of directors, CEO management and staff and financial structure.

The new Tyco International is a leading provider of fire protection, security and safety products and services, flow control products as well as electrical and metal products, with annual revenues of more than $18 billion. We are passionate about delivering quality, innovation and performance to make our customers lives easier, safer and better. Take a further look at what we've got to offer and you'll see what makes Tyco such a vital part of your world.

115,000 people
Revenue $18.2 billion (2007)
Operating in over 60 countries
Serving customers in over 100 countries
Providing thousands of vitally important products and services

Historic Timeline
1882 - Frederick Grinnell patented his automatic sprinkler
1883 - Mather + Platt purchased the rights to the Grinnell Sprinkler outside the US
1889 - Wormald Brothers distributed the Grinnell Sprinkler in Australasia on behalf of Mather + Platt
1911 - Wormald Brothers form a Limited Company selling fire protection
1960 - yco Inc formed with two primary holdings, Tyco Semiconductor and the Materials Research Laboratory
1964 - Tyco becomes a publicly owned company
1974 - Tyco acquire Simplex Technologies
1976 - Wormald acquire the Mather + Platt Group that included Atlas Fire

Engineering and Grinnell Firekil
1976 - Tyco acquires Grinnell Fire Protection Systems
1978 - Wormald acquire Ansul
1986 - Wormald Ansul (UK) Ltd is created
1989 - Wormald Engineering is created out of its parent company, Wormald Ansul (UK) Ltd.
1990 - Tyco acquire Wormald International Ltd including Wormald Ansul (UK) Ltd.
1996 - Tyco acquire Thorn Security
1997 - Tyco acquire ADT
1999 - Tyco acquire AMP
2000 - Wormald Engineering re-named to Tyco Engineering Services
2000 - Wormald Ansul (UK) Ltd. acquire Prestaroy Ltd.
2001 - Wormald Ansul (UK) Ltd. acquire Spector Lumenex Ltd. and Rhomax Engineering Ltd.
2002 - Wormald Ansul (UK) Ltd. acquire How Fire Protection
2005 - Wormald Ansul (UK) Ltd changes its name to Tyco Fire & Integrated Solutions (UK) Ltd and brings the following heritage brands under its umbrella: Grinnell Firekil, Atlas Fire, How Fire, Mather + Platt, Tyco Engineering Services, Spector Lumenex, Rhomax Engineering

STATE OF TENNESSEE
Tre Hargett, Secretary of State
Division of Business Services
William R. Snodgrass Tower
312 Rosa L. Parks AVE, 6th FL
Nashville, TN 37243-1102

Filing Information

Name: **HELENA CHEMICAL COMPANY**

General Information

Control # :	**38295**	Formation Locale:	DELAWARE
Filing Type:	Corporation For-Profit - Foreign	Date Formed:	06/27/1977
Filing Date:	06/27/1977 4:30 PM	Fiscal Year Close	12
Status:	Active		
Duration Term:	Perpetual		

Registered Agent Address
C T CORPORATION SYSTEM
STE 2021
800 S GAY ST
KNOXVILLE, TN 37929-9710
Phone:
Fax:

Principal Address
225 SCHILLING BLVD STE 300
COLLIERVILLE, TN 38017
Phone: (901) 761-005

The following document(s) was/were filed in this office on the date(s) indicated below:

Date Filed	Filing Description	Image #
03/08/2011	2010 Annual Report	6844-2279
	Principal Address 1 Changed From: 1209 ORANGE STREET To: 225 SCHILLING BLVD STE 300	
	Principal City Changed From: WILMINGTON To: COLLIERVILLE	
	Principal State Changed From: DE To: TN	
	Principal Postal Code Changed From: 198010000 To: 38017	
	Principal County Changed From: No value To: SHELBY	
03/10/2010	2009 Annual Report	6671-2511
07/28/2009	Assumed Name Renewal	6575-2081
03/04/2009	2008 Annual Report	6464-0251
03/04/2008	2007 Annual Report	6234-1815
03/09/2007	2006 Annual Report	5980-0188
02/02/2006	2005 Annual Report	5675-0965
01/24/2005	2004 Annual Report	5334-3191
09/27/2004	Registered Agent Change (by Agent)	5243-0482
	Registered Agent Physical Address Changed	

Filing Information

Name: **HELENA CHEMICAL COMPANY**

09/24/2004	Assumed Name	5242-1308
03/15/2004	2003 Annual Report	5065-0144
04/07/2003	2002 Annual Report	4786-1843
03/28/2002	2001 Annual Report	4461-1693
03/30/2001	2000 Annual Report	4163-2455
03/27/2000	1999 Annual Report	3862-3636
07/16/1999	Assumed Name Renewal	3713-2369
04/21/1997	CMS Annual Report Update	3330-0447
	Fiscal Year Close Changed	
08/12/1994	Assumed Name	2878-2363
08/12/1994	Assumed Name	2878-2365
09/24/1990	Administrative Amendment	1941-0654
	Mail Address Changed	
09/14/1990	Administrative Amendment	1930-0393
	Mail Address Changed	
06/16/1990	Administrative Amendment	FYC/REVENUE
	Fiscal Year Close Changed	
08/22/1985	Articles of Amendment	560 01695
	Shares of Stock Changed	
08/20/1985	Articles of Amendment	559 03556
	Shares of Stock Changed	
	Principal Address Changed	
08/07/1979	Registered Agent Change (by Agent)	093 00752
	Registered Agent Physical Address Changed	
	Registered Agent Changed	
08/25/1977	Articles of Amendment	FOREIGN
	Name Changed	
06/27/1977	Initial Filing	FOREIGN

Active Assumed Names (if any)	Date	Expires
SUGARTECH	09/24/2009	09/24/2014

Delaware

The First State

I, JEFFREY W. BULLOCK, SECRETARY OF STATE OF THE STATE OF DELAWARE, DO HEREBY CERTIFY THE CERTIFICATE OF MERGER, WHICH MERGES:

"ANSUL, LLC", A DELAWARE LIMITED LIABILITY COMPANY,

WITH AND INTO "TYCO FIRE PRODUCTS LP" UNDER THE NAME OF "TYCO FIRE PRODUCTS LP", A LIMITED PARTNERSHIP ORGANIZED AND EXISTING UNDER THE LAWS OF THE STATE OF DELAWARE, WAS RECEIVED AND FILED IN THIS OFFICE THE EIGHTEENTH DAY OF DECEMBER, A.D. 2009, AT 10:06 O'CLOCK P.M.

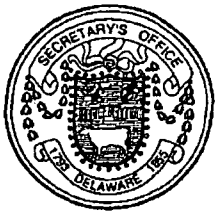
AND I DO HEREBY FURTHER CERTIFY THAT THE AFORESAID LIMITED PARTNERSHIP SHALL BE GOVERNED BY THE LAWS OF THE STATE OF DELAWARE.

AND I DO HEREBY FURTHER CERTIFY THAT THE EFFECTIVE DATE OF THE AFORESAID CERTIFICATE OF MERGER IS THE TWENTY-FIFTH DAY OF DECEMBER, A.D. 2009.

RECEIVED
FEB 16 2010
Secretary of State

4727421 8330

100119575



Jeffrey W. Bullock, Secretary of State

AUTHENTICATION: 7802798

DATE: 02-08-10

Corporation Name	TYCO INTERNATIONAL (US) INC.
Fictitious Names	
Filing #	100144851
Filing Type	Foreign For Profit Corporation
Filed under Act	For Bus Corp; 958 of 1987
Status	Merged
Principal Address	
Reg. Agent	THE CORPORATION COMPANY
Agent Address	425 WEST CAPITOL AVENUE, SUITE 1700 LITTLE ROCK, AR 72201
Date Filed	03/04/1997
Officers	SEE FILE, Incorporator/Organizer EDWARD D. BREEN, President JUDITH A. REINSDORF, Secretary EDWARD C. ARDITTE, Vice-President J. WILLIAM MCARTHUR JR., Treasurer LINDA AUGER, Controller
Foreign Name	N/A
Foreign Address	ONE TYCO PARK EXETER, 03833
State of Origin	MA

Pay Franchise Tax for this corporation

LLC Member information is now confidential per Act 865 of 2007

Use your browser's back button to return to the Search Results

Begin New Search

Roller-Citizens Funeral Home
508 East Plaza Street
West Helena, AR 72390
870-572-2571
staff.whelena@rollerfuneralhomes.com
Map & Directions

Obituary

Service Details | Guestbook

William "Bill" John Brothers, Jr.

March 11, 1920 - September 22, 2009

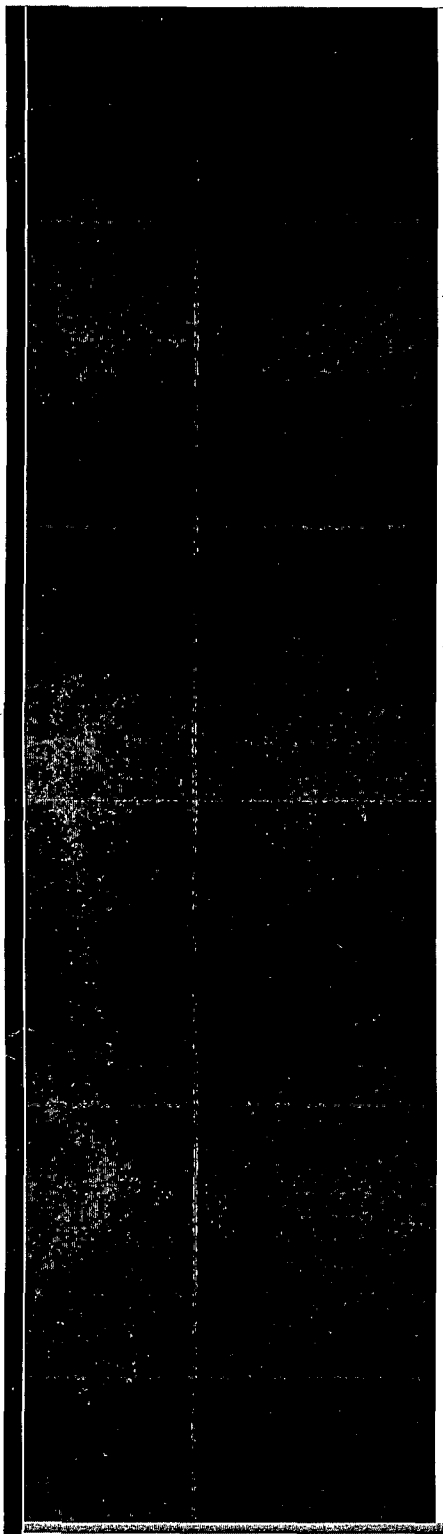
William "Bill" John Brothers, Jr.

William John Brothers, Jr., of Helena, Arkansas, businessman and family man, passed away on Tuesday, September 22, 2009 in Helena. Mr. Brothers was 89 years old, born in Shelby, Mississippi on March 11, 1920.

He was married for 59 years to Cassie Campbell Brothers. He was the son of the late William John Brothers, Sr. and Ann Kingston Brothers of North Sydney, Nova Scotia. He is survived by his wife, Cassie; two children, William John Brothers, III and his wife Suzanne, and Brooke Tappan and her husband, Charlie. Mr. Brothers is also survived by six grandchildren whom he loved dearly—Charles M. Tappan, Jr. and his wife, Lynne, Victoria Tappan, Bill Tappan, William John Brothers, IV, Kingston Brothers and Rowland Brothers. Bill was a family man and always put his family first. He was a member of St. Mary's Catholic Church in Helena, Arkansas.

Bill was preceded in death by his parents; two brothers, P.K. Brothers and Pierre Brothers; and a sister, Elsbeth McLean. He is survived by four sisters, Justine Crossley of Memphis, Tennessee, Juan Thompson of Owensboro, Kentucky, Natasha Harvey of Oklahoma City, Oklahoma, Denoysia Hume of Newport News, Virginia; two sisters-in-law, Marie Brothers of Memphis, Tennessee and Emma Lee Gordon and her husband, Al, of Helena, Arkansas; his family remaining in Canada; cousins Stanley Brothers and his wife Nora, Ken Brothers and his wife Cheryl, and his nephew, Bob McLean; and close family friend, Dr. Jim Adkins, affectionately known as Lemoyne and his wife Bess. Bill adored and enjoyed all his nieces and nephews.

Bill received his education at Christian Brothers in Memphis, Tennessee. He loved airplanes and was flying by the age of sixteen. After four years of service with the Army Air Force during WWII, he came to Helena to operate Terry Aircraft and Helena Airport. Years later he became a part of Helena Chemical Company. He established Blackhawk Warehousing and Leasing Company in 1969. He devoted the remainder of his working career to Blackhawk and its subsidiaries. He was devoted to his employees as well as to his family. They and their families were very important to him.



Bill said many times, "I can't help where I was born, but I know where I'll be buried." He dearly loved Arkansas and in particular, Phillips County. He served on the Arkansas State Police Commission (the best troopers in the world), and served 28 years on the Board of Trustees of Phillips College of the University of Arkansas. He was awarded their first honorary degree. He was active in the local Chamber of Commerce, Helena Rotary Club and served as treasurer of the Arkansas State Chamber of Commerce. He chaired the Phillips County Industrial Development Corporation and was recipient of award for Exceptional Accomplishment for Arkansas Community Development Program in 1975. He served on the Civil Service Commission and the Welfare Board. He received the Citizen of the Year award in 1975.

Bill was a frank and honest man. He always talked straight even if it hurt.

Bill, a Staff Sergeant—Aerial Engineer, served as a non-commissioned officer in charge of flight test while in the military and received the Air Medal for Heroism: Soldiers Medal: Fidelity Efficiency Honors: American Defense: American Campaign: World War II: European African Middle Eastern Campaign.

Memorials may be made to St. Mary's Catholic Church, 123 Columbia, Helena, Arkansas 72342, or the charity of your choice.

Services for Bill Brothers will be held at 10 a.m., Saturday, September, 26, 2009 at St. Mary's Catholic Church in Helena. Visitation will be Friday evening beginning at 5:30 p.m. until 9 p.m., with a rosary service at 6:30 p.m. at Roller-Citizens. Burial will be at Maple Hill Cemetery in Helena.

Pallbearers are Buddy Formby, Jerry Daughtery, Charles M. Tappan, Jr., Bill Tappan, John Brothers and Gill Pillow.

Honorary pallbearers are Billy Mitchell, Chance Stokes, David Solomon, Jim Howe, Tim Owens, Chris Carnathan, Jeff Carnathan and Charlie Blue.

Services will be directed by Roller-Citizens Funeral Home, West Helena, (870) 572-2571.

Print Obituary

Service Details *Guestbook*

Back